1/7/1
DIALOG(R)File 351: Derwent WPI

0014235816
WPI Acc no: 2004-421776/200440
XRAM Acc no: C2004-158565
Powdered formulation used e.g. as food additive or pharmaceutical for preventing arteriosclerosis and hyperlipemia, contains phytosterol
Patent Assignee: BASF AG (BADI)
Inventor: AUWETER H; BOHN H; HASSELWANDER O; RUNGE F

Patent Family (9 patents, 106 countries)

Patent Number	Kind	Date	Application Number	Kind	Date	Update	Type
DE 10253111	A1	20040527	DE 10253111	A	20021113	200440	B
WO 2005009144	A1	20050203	WO 2003EP12557	A	20031111	200510	E
AU 2003304369	A1	20050214	AU 2003304369	A	20031111	200532	E
EP 1575378	A1	20050921	EP 2003817484	A	20031111	200562	E
			WO 2003EP12557	A	20031111		
US 20060035871	A1	20060216	WO 2003EP12557	A	20031111	200614	E
			US 2005534543	A	20050510		
JP 2006514829	W	20060518	WO 2003EP12557	A	20031111	200635	E
			JP 2005504531	A	20031111		
CN 1741748	A	20060301	CN 200380103173	A	20031111	200649	E
NZ 540473	A	20070126	NZ 540473	A	20031111	200711	E
			WO 2003EP12557	A	20031111		
IN 200501225	P4	20070727	WO 2003EP12557	A	20031111	200770	E
			IN 2005CN1225	A	20050613		

Priority Applications (no., kind, date): DE 10253111 A 20021113

Patent Details

Patent Number	Kind	Lan	Pgs	Draw	Filing Notes
DE 10253111	A1	DE	7	0	
WO 2005009144	A1	DE			
National Designated States,Original	AE AG AL AM AT AU AZ BA BB BG BR BW BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE EG ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PG PH PL PT RO RU SC SD SE SG SK SL SY TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW				
Regional Designated States,Original	AT BE BG BW CH CY CZ DE DK EA EE ES FI FR GB GH GM GR HU IE IT KE LS LU MC MW MZ NL OA PT RO SD				

	SE SI SK SL SZ TR TZ UG ZM ZW				
AU 2003304369	A1	EN		Based on OPI patent	WO 2005009144
EP 1575378	A1	DE		PCT Application	WO 2003EP12557
				Based on OPI patent	WO 2005009144
Regional Designated States,Original	AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LT LU LV MC MK NL PT RO SE SI SK TR				
US 20060035871	A1	EN		PCT Application	WO 2003EP12557
JP 2006514829	W	JA	17	PCT Application	WO 2003EP12557
				Based on OPI patent	WO 2005009144
NZ 540473	A	EN		PCT Application	WO 2003EP12557
				Based on OPI patent	WO 2005009144
IN 200501225	P4	EN		PCT Application	WO 2003EP12557

Alerting Abstract DE A1
NOVELTY - Powdered formulation (A) contains at least one phytosterol (I) and has a mean particle size of 0.01-100 mum.
DESCRIPTION - An INDEPENDENT CLAIM is also included for preparation of (A).
ACTIVITY - Antiarteriosclerotic; Antilipemic.
No biological data is given.
MECHANISM OF ACTION - None given.
USE - (A) are used to prepare nutritional supplements, and as additives for foods or animal feeds and as pharmaceuticals or cosmetic compositions. (I) are known for prevention of arteriosclerosis and hyperlipemia and reduce serum cholesterol levels.
ADVANTAGE - (A) can be incorporated into both aqueous and oily formulations.

Technology Focus
PHARMACEUTICALS - Preferred Composition: At least one (I) is present in partially amorphous form, optionally embedded in a protective colloid (PC) matrix. (A) contains (in wt.%) 0.1-80 (preferably 5-25) (I) (dry basis), 5-70 (preferably 10-60) at least one PC, 0.1-70 (preferably 20-50) at least one plasticizer 0.01-70 (preferably 0.5-20) at least one emulsifier and 0.01-50 (preferably 1) at least one antioxidant and/or preservative. (A) is water dispersible and optionally also contains carotenoids and vitamins and has a mean particle size of 0.05-1 mum.
Preferred Compounds: (I) are stigmasterol (Ia), campesterol (Ib) and beta-sitosterol (Ic), or their hydrogenated derivatives, particularly a mixture produced by distillation of soya oil and containing (in wt.%): 40-55 (Ic), 20-30 (Ib) and 14-22 (Ia).

ORGANIC CHEMISTRY - Preparation: At least one (I) is dissolved in a water miscible organic solvent (optionally mixed with water) or a water-immiscible solvent, then the solution is mixed with an aqueous, molecularly or colloidally dispersed PC, and the resulting dispersion (in which (I) is the dispersed phase) is freed of solvent and water to form a dry powder. This is dried, optionally in the presence of a coating material. Particularly (I) is dissolved in water-miscible solvent at 50-240 (preferably 140-180)(deg)C and the solution is mixed with the PC component at 35-80(deg)C. Alternately, at least one (I) is milled in aqueous medium in the presence of PC and the resulting suspension dried to give (A). Milling is particularly at a temperature high enough to cause (partial) melting of (I), and the melt is cooled before conversion to powder. Especially milling is for 0.05-200 seconds at 150-200(deg)C, with subsequent cooling to 20-80(deg)C.
Preferred Materials: The PC comprises pectin, casein, caseinate, gum arabic, modified starch or fish

gelatin. Plasticizers are e.g. sucrose, glucose, lactose or sorbitol and emulsifiers are long-chain fatty esters of ascorbic acid, esters of mono-fatty acid glycerides with acetic, citric or lactic acids or lecithin, preferably ascorbyl palmitate.

Title Terms /Index Terms/Additional Words: POWDER; FORMULATION; FOOD; ADDITIVE; PHARMACEUTICAL; PREVENT; ARTERIOSCLEROSIS; CONTAIN; PHYTOSTEROL

Class Codes

International Patent Classification

IPC	Class Level	Scope	Position	Status	Version Date
A23L-001/30			Main		"Version 7"
A23K-001/165; A61K-031/575			Secondary		"Version 7"
A23K-0001/00	A	I		R	20060101
A23K-0001/16	A	I		R	20060101
A23K-0001/165	A	I	L	B	20060101
A23L-0001/30	A	I		R	20060101
A23L-0001/30	A	I	F	B	20060101
A61K-0031/56	A	I	F	B	20060101
A61K-0031/575	A	I	L	B	20060101
A61K-0031/575	A	I		R	20060101
A61K-0008/63	A	I		R	20060101
A61K-0009/14	A	I	L	B	20060101
A61P-0003/02	A	I	L	B	20060101
A61P-0003/06	A	I	L	B	20060101
A61Q-0001/00	A	I		R	20060101
A61Q-0019/00	A	I		R	20060101
A23K-0001/00	C	I		R	20060101
A23K-0001/16	C	I		R	20060101
A23L-0001/30	C	I		R	20060101
A23L-0001/30	C	I	L	B	20060101
A61K-0031/56	C	I	L	B	20060101
A61K-0031/575	C	I		R	20060101
A61K-0008/30	C	I		R	20060101
A61P-0003/00	C	I	L	B	20060101
A61Q-0001/00	C	I		R	20060101
A61Q-0019/00	C	I		R	20060101

US Classification, Issued: 514169000, 424442000

File Segment: CPI
DWPI Class: A96; A97; B07; D13; D21

Manual Codes (CPI/A-N): A12-V01; A12-W09; B01-D02; B03-F; B04-B04K; B04-C02B; B04-C02D; B04-N02; B07-A02; B10-A07; B12-M11G; B14-D02A2; B14-F06; B14-F07; D03-G; D03-H01T2 ; D08-B

Original Publication Data by Authority

Australia

Publication No. AU 2003304369 A1 (Update 200532 E)
Publication Date: 20050214
PULVERULENT PHYTOSTEROL FORMULATIONS
Assignee: BASF AG (BADI)
Inventor: AUWETER H
BOHN H
HASSELWANDER O
RUNGE F
Language: EN
Application: AU 2003304369 A 20031111 (Local application)
Priority: DE 10253111 A 20021113
Related Publication: WO 2005009144 A (Based on OPI patent)
Original IPC: A23L-1/30(A) A23K-1/165(B) A61K-31/575(B)
Current IPC: A23K-1/00(R,A,I,M,EP,20060101,20051008,A) A23K-1/00 (R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16 (R,I,M,EP,20060101,20051008,C) A23L-1/30(R,I,M,EP,20060101,20051008,A) A23L-1/30 (R,I,M,EP,20060101,20051008,C) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575 (R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63 (R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00 (R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00 (R,I,M,EP,20060101,20060722,C)

China

Publication No. CN 1741748 A (Update 200649 E)
Publication Date: 20060301
Assignee: BASF AG; DE (BADI)
Inventor: AUWETER H
BOHN H
HASSELWANDER O
RUNGE F
Language: ZH
Application: CN 200380103173 A 20031111 (Local application)
Priority: DE 10253111 A 20021113
Original IPC: A23K-1/165(I,CN,20060101,A,L) A23L-1/30(I,CN,20060101,A,F) A61K-31/575 (I,CN,20060101,A,L) A61K-8/30(I,98,20060101,C,L) A61K-8/63(I,CN,20060101,A,L)
Current IPC: A23K-1/00(R,A,I,M,EP,20060101,20051008,A) A23K-1/00 (R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16 (R,I,M,EP,20060101,20051008,C) A23L-1/30(B,I,H,CN,20060101,20060301,A,F) A23L-1/30 (B,I,H,CN,20060101,20060301,C,L) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575

(R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63 (R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00 (R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00 (R,I,M,EP,20060101,20060722,C)

Germany

Publication No. DE 10253111 A1 (Update 200440 B)
Publication Date: 20040527
Pulverformige Phytosterol-Formulierungen
Assignee: BASF AG, 67063 Ludwigshafen, DE (BADI)
Inventor: Auweter, Helmut, Dr., 67117 Limburgerhof, DE
Bohn, Heribert, 67319 Wattenheim, DE
Hasselwander, Oliver, Dr., 76829 Landau, DE
Runge, Frank, Dr., 67159 Friedelsheim, DE
Language: DE (7 pages, 0 drawings)
Application: DE 10253111 A 20021113 (Local application)
Original IPC: C07J-9/00(A) A23K-1/00(B) A23L-1/30(B) A61K-7/00(B)
Current IPC: A23K-1/00(R,A,I,M,EP,20060101,20051008,A) A23K-1/00 (R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16 (R,I,M,EP,20060101,20051008,C) A23L-1/30(R,I,M,EP,20060101,20051008,A) A23L-1/30 (R,I,M,EP,20060101,20051008,C) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575 (R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63 (R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00 (R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00 (R,I,M,EP,20060101,20060722,C)
Original Abstract:
Die Erfindung betrifft pulverformige Phytosterol-Formulierungen, Verfahren zu ihrer Herstellung sowie ihre Verwendung.

Claim:

1. Pulverformige Phytosterol-Formulierungen, enthaltend mindestens ein Phytosterol mit einer mittleren Teilchengrosse von 0,01 bis 100 mum.

EPO

Publication No. EP 1575378 A1 (Update 200562 E)
Publication Date: 20050921
PULVERFORMIGE PHYTOSTEROL-FORMULIERUNGEN
PULVERULENT PHYTOSTEROL FORMULATIONS
FORMULATIONS PULVERULENTES A BASE DE PHYTOSTEROLS
Assignee: BASF Aktiengesellschaft, 67056 Ludwigshafen, DE (BADI)
Inventor: AUWETER, Helmut, Lessingstr. 35, 67117 Limburgerhof, DE
BOHN, Heribert, Jakob-Ries-Strasse 10, 67319 Wattenheim, DE
HASSELWANDER, Oliver, Hamburger Strasse 52, 76829 Landau, DE
RUNGE, Frank, Am Tiergarten 7, 67159 Friedelsheim, DE
Language: DE

Application: EP 2003817484 A 20031111 (Local application)
WO 2003EP12557 A 20031111 (PCT Application)
Priority: DE 10253111 A 20021113
Related Publication: WO 2005009144 A (Based on OPI patent)
Designated States: (Regional Original) AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LT LU LV MC MK NL PT RO SE SI SK TR
Original IPC: A23L-1/30(A) A61K-31/575(B)
Current IPC: A23K-1/00(R,A,I,M,EP,20060101,20051008,A) A23K-1/00 (R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16 (R,I,M,EP,20060101,20051008,C) A23L-1/30(R,I,M,EP,20060101,20051008,A) A23L-1/30 (R,I,M,EP,20060101,20051008,C) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575 (R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63 (R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00 (R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00 (R,I,M,EP,20060101,20060722,C)
Original Abstract:
The invention relates to pulverulent phytosterol formulations, methods for the production and use thereof. The aim of said invention is to deliver the phytosterol formulations incorporatable in aqueous and oily preparations. For this purpose, said pulverulent phytosterol formulations contain at least one type of phytosterol whose mean particle size ranges from 0.01 and 100 mum, preferably from 0.01 to 10 mum, better from 0.01 and 2 mum and ideally from 0.05 and 1 mum. Said phytosterol formulations are characterised, in particular in that at least one type of phytosterol is used in a semi-amorphous form. In a preferred embodiment, said phytosterol formulations are characterised in that said phytosterol is embedded in a protective colloid matrix.

India

Publication No. IN 200501225 P4 (Update 200770 E)
Publication Date: 20070727
Language: EN
Application: WO 2003EP12557 A 20031111 (PCT Application)
IN 2005CN1225 A 20050613 (Local application)
Priority: DE 10253111 A 20021113
Original IPC: A23L-1/30(A)
Current IPC: A23L-1/30(A)

Japan

Publication No. JP 2006514829 W (Update 200635 E)
Publication Date: 20060518
Language: JA (17 pages)
Application: WO 2003EP12557 A 20031111 (PCT Application)
JP 2005504531 A 20031111 (Local application)
Priority: DE 10253111 A 20021113
Related Publication: WO 2005009144 A (Based on OPI patent)
Original IPC: A23K-1/165(B,I,H,JP,20060101,20060414,A,L) A23L-1/30 (B,I,H,JP,20060101,20060414,A,F) A61K-31/575(B,I,H,JP,20060101,20060414,A,L) A61K-9/14 (B,I,H,JP,20060101,20060414,A,L) A61P-3/00(B,I,H,98,20060101,20060414,C,L) A61P-3/02 (B,I,H,JP,20060101,20060414,A,L) A61P-3/06(B,I,H,JP,20060101,20060414,A,L)

Current IPC: A23K-1/165(B,I,H,JP,20060101,20060414,A,L) A23L-1/30
(B,I,H,JP,20060101,20060414,A,F) A61K-31/575(B,I,H,JP,20060101,20060414,A,L) A61K-9/14
(B,I,H,JP,20060101,20060414,A,L) A61P-3/00(B,I,H,98,20060101,20060414,C,L) A61P-3/02
(B,I,H,JP,20060101,20060414,A,L) A61P-3/06(B,I,H,JP,20060101,20060414,A,L)

New Zealand

Publication No. NZ 540473 A (Update 200711 E)
Publication Date: 20070126
Assignee: BASF AG (BADI)
Inventor: AUWETER H
BOHN H
HASSELWANDER O
RUNGE F
Language: EN
Application: NZ 540473 A 20031111 (Local application)
WO 2003EP12557 A 20031111 (PCT Application)
Priority: DE 10253111 A 20021113
Related Publication: WO 2005009144 A (Based on OPI patent)
Original IPC: A23L-1/30(A) A23K-1/165(B) A61K-31/575(B)
Current IPC: A23L-1/30(A) A23K-1/165(B) A61K-31/575(B)

United States

Publication No. US 20060035871 A1 (Update 200614 E)
Publication Date: 20060216
Pulverulent phytosterol formulations
Assignee: BASF Aktiengesellschaft, Ludwigshafen 67056, DE (BADI)
Auweter, Helmut, Lessingstr. 35, Limburgerhof 67117, DE Residence: DE Nationality: DE
Bohn, Heribert, Wattenheim, DE Residence: DE Nationality: DE
Hasselwander, Oliver, Landau, DE Residence: DE Nationality: DE
Runge, Frank, Friedelsheim, DE Residence: DE Nationality: DE
Inventor: Auweter, Helmut, Lessingstr. 35, Limburgerhof 67117, DE Residence: DE Nationality: DE
Bohn, Heribert, Wattenheim, DE Residence: DE Nationality: DE
Hasselwander, Oliver, Landau, DE Residence: DE Nationality: DE
Runge, Frank, Friedelsheim, DE Residence: DE Nationality: DE
Agent: OBLON, SPIVAK, MCCLELLAND, MAIER & NEUSTADT, P.C., 1940 DUKE STREET, ALEXANDRIA, VA, US
Language: EN
Application: WO 2003EP12557 A 20031111 (PCT Application)
US 2005534543 A 20050510 (Local application)
Priority: DE 10253111 A 20021113
Original IPC: A61K-31/56(B,I,H,US,20060101,20060216,A,F)
Current IPC: A23K-1/00(R,I,M,EP,20060101,20051008,A) A23K-1/00
(R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16
(R,I,M,EP,20060101,20051008,C) A23L-1/30(R,I,M,EP,20060101,20051008,A) A23L-1/30
(R,I,M,EP,20060101,20051008,C) A61K-31/56(B,I,H,US,20060101,20060216,A,F) A61K-31/56
(B,I,H,US,20060101,20060216,C,L) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575
(R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63
(R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00

(R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00 (R,I,M,EP,20060101,20060722,C)
Original US Class (secondary): 514169 424442
Original Abstract:
A pulverulent phytosterol formulation comprising at least one phytosterol having a mean particle size ranging from 0.01 to 100 mum, a process for producing the pulverulent phytosterol formulation, and a food supplement, an animal feed, food or pharmaceutical or cosmetic preparation comprising the phytosterol formulation.

Claim:

1. 1. A pulverulent phytosterol formulation comprising at least one phytosterol having a mean particle size of from 0.01 to 100 mum.

WIPO

Publication No. WO 2005009144 A1 (Update 200510 E)
Publication Date: 20050203
PULVERFORMIGE PHYTOSTEROL-FORMULIERUNGEN
PULVERULENT PHYTOSTEROL FORMULATIONS
FORMULATIONS PULVERULENTES A BASE DE PHYTOSTEROLS
Assignee: *(except US)* BASF AKTIENGESELLSCHAFT, 67056 Ludwigshafen, DE Residence: DE Nationality: DE (BADI)
(only US) AUWETER, Helmut, Lessingstr. 35, 67117 Limburgerhof, DE Residence: DE Nationality: DE
(only US) BOHN, Heribert, Jakob-Ries-Strasse 10, 67319 Wattenheim, DE Residence: DE Nationality: DE
(only US) HASSELWANDER, Oliver, Hamburger Strasse 52, 76829 Landau, DE Residence: DE Nationality: DE
(only US) RUNGE, Frank, Am Tiergarten 7, 67159 Friedelsheim, DE Residence: DE Nationality: DE
Inventor: AUWETER, Helmut, Lessingstr. 35, 67117 Limburgerhof, DE Residence: DE Nationality: DE
BOHN, Heribert, Jakob-Ries-Strasse 10, 67319 Wattenheim, DE Residence: DE Nationality: DE
HASSELWANDER, Oliver, Hamburger Strasse 52, 76829 Landau, DE Residence: DE Nationality: DE
RUNGE, Frank, Am Tiergarten 7, 67159 Friedelsheim, DE Residence: DE Nationality: DE
Agent: BASF AKTIENGESELLSCHAFT, 67056 Ludwigshafen, DE
Language: DE
Application: WO 2003EP12557 A 20031111 (Local application)
Priority: DE 10253111 A 20021113
Designated States: (National Original) AE AG AL AM AT AU AZ BA BB BG BR BW BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE EG ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PG PH PL PT RO RU SC SD SE SG SK SL SY TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW
(Regional Original) AT BE BG BW CH CY CZ DE DK EA EE ES FI FR GB GH GM GR HU IE IT KE LS LU MC MW MZ NL OA PT RO SD SE SI SK SL SZ TR TZ UG ZM ZW
Original IPC: A23L-1/30(A) A23K-1/165(B) A61K-7/00(B) A61K-31/575(B)
Current IPC: A23K-1/00(R,A,I,M,EP,20060101,20051008,A) A23K-1/00

(R,I,M,EP,20060101,20051008,C) A23K-1/16(R,I,M,EP,20060101,20051008,A) A23K-1/16
(R,I,M,EP,20060101,20051008,C) A23L-1/30(R,I,M,EP,20060101,20051008,A) A23L-1/30
(R,I,M,EP,20060101,20051008,C) A61K-31/575(R,I,M,EP,20060101,20051008,A) A61K-31/575
(R,I,M,EP,20060101,20051008,C) A61K-8/30(R,I,M,EP,20060101,20060722,C) A61K-8/63
(R,I,M,EP,20060101,20060722,A) A61Q-1/00(R,I,M,EP,20060101,20060722,A) A61Q-1/00
(R,I,M,EP,20060101,20060722,C) A61Q-19/00(R,I,M,EP,20060101,20060722,A) A61Q-19/00
(R,I,M,EP,20060101,20060722,C)

Original Abstract:
Die Erfindung betrifft pulverformige Phytosterol-Formulierungen, Verfahren zu ihrer Herstellung sowie ihre Verwendung. Aufgabe der vorliegenden Erfindung war es, Phytosterol-haltige Formulierungen bereitzustellen, die sowohl in wassrige als auch in olige Zubereitungen eingearbeitet werden konnen. Diese Aufgabe wurde erfindungsgemass gelost durch pulverformige Phytosterol-Formulierungen, enthaltend mindestens ein Phytosterol mit einer mittleren Teilchengrosse im Bereich von 0,01 bis 100 microm, bevorzugt im Bereich von 0,01 bis 2 microm, ganz besonders bevorzugt im Bereich von 0,05 bis 1 microm. Die erfindungsgemassen Phytosterol-Formulierungen sind u.a. auch dadurch gekennzeichnet, dass mindestens ein Phytosterol in teilamorpher Form vorliegt. Eine weitere bevorzugte Ausfuhrungsform der Phytosterol-Formulierungen ist dadurch gekennzeichnet, dass das Phytosterol in einer Schutzkolloid-Matrix eingebettet ist.

The invention relates to pulverulent phytosterol formulations, methods for the production and use thereof. The aim of said invention is to deliver the phytosterol formulations incorporatable in aqueous and oily preparations. For this purpose, said pulverulent phytosterol formulations contain at least one type of phytosterol whose mean particle size ranges from 0,01 and 100 mum, preferably from 0,01 to 10 mum, better from 0,01 and 2 mum and ideally from 0,05 and 1 mum. Said phytosterol formulations are characterised, in particular in that at least one type of phytosterol is used in a semi-amorphous form. In a preferred embodiment, said phytosterol formulations are characterised in that said phytosterol is embedded in a protective colloid matrix.

L'invention concerne des formulations pulverulentes a base de phytosterols, des procedes de production de ces formulations et l'utilisation desdites formulations. L'objectif de l'invention est de fournir des formulations a base de phytosterols pouvant etre incorporees aussi bien dans des preparations aqueuses que dans des preparations huileuses. A cet effet, les formulations pulverulentes a base de phytosterols selon l'invention contiennent au moins un phytosterol presentant une taille moyenne de particules comprise entre 0,01 et 100 mum, de preference entre 0,01 et 10 mum, mieux encore entre 0,01 et 2 mum et idealement entre 0,05 et 1 mum. Ces formulations a base de phytosterols se caracterisent notamment en ce qu'au moins un phytosterol est present sous forme semi-amorphe. Un autre mode de realisation prefere de ces formulations a base de phytosterols se caracterise en ce que le phytosterol est enrobe dans une matrice colloidale de protection.

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(54) Bezeichnung: **Pulverförmige Phytosterol-Formulierungen**

(57) Zusammenfassung: Die Erfindung betrifft pulverförmige Phytosterol-Formulierungen, Verfahren zu ihrer Herstellung sowie ihre Verwendung.

Beschreibung

[0001] Die Erfindung betrifft pulverförmige Phytosterol-Formulierungen, Verfahren zu ihrer Herstellung und deren Verwendung in Nahrungsergänzungsmitteln, in Lebensmitteln und Tierfuttermitteln sowie in pharmazeutischen und kosmetischen Präparaten.

[0002] Als Phytosterole werden Sterole bezeichnet, die aus Pflanzen und Hefen isoliert werden. Die wichtigsten Vertreter dieser Stoffklasse sind z.B. Stigmasterol, Campesterol und β-Sitosterol sowie deren hydrierte Derivate wie Campestanol und β-Sitostanol. Phytosterole sind strukturell dem Cholesterol ähnlich. Da beispielsweise β-Sitosterol die Absorption von Cholesterol hemmt, wird es als Lipidsenker zur Prophylaxe von Arteriosklerose und Hyperlipidämie eingesetzt.

[0003] Zur Senkung des Cholesterinspiegels werden Phytosterole häufig als Zusatzstoffe in diätetischen Lebensmitteln wie z.B. für Margarine verwendet.

[0004] Phytosterole sind in Wasser unlöslich, während in Fetten und Ölen eine nur geringe Löslichkeit gefunden wird. Diese begrenzte Löslichkeit erschwert häufig die Anwendbarkeit der Phytosterole bei der Herstellung von Lebensmittelzubereitungen sowie von kosmetischen Produkten. Unzureichende Wirksamkeiten einerseits sowie eine schlechte Dispergierbarkeit in kosmetischen und Lebensmittelzubereitungen andererseits resultieren häufig aus den schlechten Löslichkeiten der Phytosterole.

[0005] Verschiedene Verfahren zur Herstellung Phytosterol-haltiger Formulierungen sind bereits bekannt. So beschreibt EP-A-0 289 636 Solubilisate von Phytosterolen in einer wässrigen Lösung von Polyhydroxyverbindungen oder Sucrose-Fettsäureestern.

[0006] Weitere flüssige Zubereitungen von Phytosterolen zusammen mit Solubilisatoren sind offenbart in US 3,865,939 und US 5,244,887.

[0007] EP-A-1 197 153 beschreibt wässrige Dispersionen oder Suspensionen von Phytosterolen in Gegenwart von nicht-sterolartigen Emulgatoren und deren Verwendung in Lebensmitteln, beispielweise in Brotaufstrichen.

[0008] WO 01/37681 betrifft wässrige Phytosterol-haltige Zusammensetzungen, erhältlich durch Homogenisieren von Phytosterolen in Wasser in Gegenwart eines wasserlöslichen Proteins, beispielsweise in Gegenwart von Casein und daraus hergestellte wasserdispergierbare Pulver.

[0009] Aufgabe der vorliegenden Erfindung war es, Phytosterol-haltige Formulierungen bereitzustellen, die sowohl in wässrige als auch in ölige Zubereitungen eingearbeitet werden können.

[0010] Diese Aufgabe wurde erfindungsgemäß gelöst durch pulverförmige Phytosterol-Formulierungen, enthaltend mindestens ein Phytosterol mit einer mittleren Teilchengröße im Bereich von 0,01 bis 100 µm, bevorzugt im Bereich von 0,01 bis 10 µm, besonders bevorzugt im Bereich von 0,01 bis 2 µm, ganz besonders bevorzugt im Bereich von 0,05 bis 1 µm.

[0011] Als Phytosterole sind im Rahmen der Erfindung bevorzugt die drei Verbindungen Stigmasterol, Campesterol und β-Sitosterol sowie deren hydrierte Derivate Stigmastanol, Campestanol und β-Sitostanol gemeint. Besonders bevorzugt sind die durch Destillation aus Sojaöl gewonnenen Phytosterol Mischungen, die im wesentlichen aus Stigmasterol, Campesterol und β-Sitosterol bestehen.

[0012] Eine typische aus Pflanzenölen gewonnene Mischung dieser drei Phytosterole besteht aus ca. 40 bis 58 Gew.-% β-Sitosterol, 20 bis 30 Gew.-% Campesterol und 14 bis 22 Gew.-% Stigmasterol.

[0013] Die erfindungsgemäßen Phytosterol-Formulierungen sind u.a. auch dadurch gekennzeichnet, dass mindestens ein Phytosterol in teilamorpher Form vorliegt.

[0014] Der Kristallinitätsgrad der Phytosterole in den erfindungsgemäßen Formulierungen läßt sich beispielsweise durch Röntgenbeugungsmessungen bestimmen und liegt im allgemeinen im Bereich kleiner 80 %, bevorzugt im Bereich von 30 bis 80 %, besonders bevorzugt im Bereich von 50 bis 80 %.

[0015] Eine weitere bevorzugte Ausführungsform der Phytosterol-Formulierungen ist dadurch gekennzeichnet, dass das Phytosterol in einer Schutzkolloid-Matrix eingebettet ist.

[0016] Geeignete Schutzkolloide sind sowohl elektrisch geladene Polymere (Polyelektrolyte) als auch neutrale Polymere. Typische Beispiele sind u.a. Gelatine wie Rinder-, Schweine- oder Fischgelatine, Stärke, modifizierte Stärke wie Octenylsuccinat Stärke, Dextrin, Pflanzenproteine wie Sojaproteine, die gegebenenfalls hydrolysiert sein können, Pektin, Guar gum, Xanthan, Gummi-Arabikum, Kasein, Natrium-Kaseinat, Lignin-sulfonat oder Mischungen davon. Es können aber auch Methylcellulose, Carboxymethylcellulose, Hydroxypropylcellulose, Hydroxypropylmethylcellulose, Blätterschellack und Alginate eingesetzt werden. Weiterhin eignen sich Homo- und Copolymere auf Basis von neutralen, kationischen oder anionischen Monomeren wie z.B. Ethylenoxid, Propylenoxid, Acrylsäure, Maleinsäureanhydrid, Milchsäure, N-Vinylpyrrolidon, Vinylacetat, α- und β-Asparaginsäure. Bezüglich näherer Einzelheiten wird auf R.A. Morton, Fat Soluble Vitamins, Intern. Encyclopedia of Food and Nutrition, Bd.9, Pergamon Press 1970, S. 128-131, verwiesen.

[0017] Bevorzugte Schutzkolloide sind Verbindungen, ausgewählt aus der Gruppe, bestehend aus Gelatine wie Rinder-, Schweine- und Fischgelatine, Pflanzenproteine, Pektin, Kasein, Natrium-Kaseinat, Gummi Arabicum und modifizierte Stärke. Besonders bevorzugt eingesetzte Schutzkolloide sind Pektin, Kasein, Natrium-Kaseinat, Gummi Arabicum, modifizierte Stärke und/oder Fischgelatine.

[0018] Der Phytosterolgehalt in den erfindungsgemäßen Formulierungen liegt im Bereich von 0,1 bis 80 Gew.-%, bevorzugt von 1 bis 60 Gew.-%, beson-

ders bevorzugt von 3 bis 35 Gew.-%, ganz besonders bevorzugt im Bereich von 5 bis 25 Gew.-%, wobei sich die Gew.-Prozentangaben auf die Trockenmasse des Pulvers beziehen.

[0019] Die Menge an verwendeten Schutzkolloiden liegt im Bereich von 0,1 bis 80 Gew.-%, bevorzugt 5 bis 70 Gew.-%, besonders bevorzugt im Bereich von 10 bis 60 Gew.-%. Die Gewichtsprozentangaben beziehen 1 sich auf die Trockenmasse der Phytosterol-Formulierung.

[0020] Zusätzlich können die Phytosterol-Formulierungen noch einen oder mehrere Weichmacher zur Erhöhung der mechanischen Stabilität der Pulver enthalten. Geeignete Weichmacher sind beispielsweise Zukker und Zuckeralkohole wie Saccharose Glukose, Laktose, Invertzucker, Sorbit, Mannit, Xylit oder Glycerin. Die Weichmacher können in Mengen von 0,1 bis 70 Gew.-%, bevorzugt 10 bis 60 Gew.-%, besonders bevorzugt 20 bis 50 Gew.-%, bezogen auf die Trockenmasse der Phytosterol-Formulierungen enthalten sein.

[0021] Ferner können die Formulierungen einen oder mehrere niedermolekulare oberflächenaktive Verbindungen (Emulgatoren) in einer Konzentration von 0,01 bis 70 Gew.-%, vorzugsweise 0,1 bis 50 Gew.-%, besonders bevorzugt 0,5 bis 20 Gew.-%, bezogen auf die Trockenmasse der Phytosterol-Formulierungen enthalten. Als solche eignen sich vor allem amphiphile Verbindungen oder Gemische solcher Verbindungen. Grundsätzlich kommen alle lebensmittel- oder futtermitteltauglichen sowie pharmakologisch und dermatologisch unbedenklichen Tenside mit einem HLB-Wert von 5 bis 20 in Betracht. Als entsprechende oberflächenaktive Substanzen kommen beispielsweise in Betracht: Ester langkettiger Fettsäuren mit Ascorbinsäure, Mono- und Diglyceride von Fettsäuren und deren Oxyethylierungsprodukte, Ester von Monofettsäureglyceriden mit Essigsäure, Zitronensäure, Milchsäure oder Diacetylweinsäure, Polyglycerinfettsäureester wie z.B. das Monostearat des Triglycerins, Sorbitanfettsäureester, Propylenglykolfettsäureester und Lecithin. Bevorzugt wird Ascorbylpalmitat eingesetzt.

[0022] Weiterhin können die Formulierungen noch einen oder mehrere niedermolekulare Stabilisatoren wie Antioxidantien und/oder Konservierungsmittel enthalten. Geeignete Antioxidantien oder Konservierungsmittel sind beispielsweise α-Tocopherol, Ascorbinsäure, tert.-Butyl-hydroxytoluol, tert.-Butylhydroxyanisol, Lecithin, Ethoxyquin, Methylparaben, Propylparaben, Sorbinsäure oder Natriumbenzoat. Die Antioxidantien bzw. Konservierungsstoffe können in Mengen von 0,01 bis 50 Gew.-%, bevorzugt 0,1 bis 30 Gew.-%, besonders bevorzugt 0,5 bis 20 Gew.-%, ganz besonders bevorzugt 1 bis 10 Gew.-%, bezogen auf die Trockenmasse der Phytosterol-Formulierungen, vorliegen.

[0023] Neben den Phytosterolen können die erfindungsgemäßen Formulierungen zusätzlich noch Carotinoide und Vitamine enthalten. Beispiele für Carotinoide sind u.a. β-Carotin, Bixin, Zeaxanthin, Cryptoxanthin, Citranaxanthin, Canthaxanthin, β-Apo-4-carotinal, β-Apo-8-carotinal, β-Apo-8-carotinsäureester, Astaxanthin, Lycopin oder Lutein, einzeln oder als Mischung.

[0024] Von den Vitaminen sind bevorzugt fettlösliche Vitamine wie Vitamin E, Vitamin E-Derivate z.B. Tocopherylacetat oder Tocopherylpalmitat sowie die K-Vitamine, Vitamin A und Derivate z.B. Vitamin A-Acetat, Vitamin A-Propionat oder Vitamin A-Palmitat, Vitamin D_2 und Vitamin D_3 und Mischungen zu verstehen. Die Bezeichnung Vitamin E steht in diesem Zusammenhang für natürliches oder synthetisches α-, β-, γ- oder δ-Tocopherol, bevorzugt für natürliches oder synthetisches α-Tocopherol sowie für Tocotrienol.

[0025] Die erfindungsgemäßen Phytosterol-Formulierungen zeichnen sich u.a. dadurch aus, dass sie sowohl in öligen als auch in wässrigen Systemen, beispielsweise in Getränken gut dispergierbar sind.

[0026] Gegenstand der Erfindung ist auch ein Verfahren zur Herstellung der oben beschriebenen pulverförmigen Phytosterol-Formulierungen, dadurch gekennzeichnet, dass man

a_1) ein oder mehrere Phytosterole in einem mit Wasser mischbaren, organischen Lösungsmittel oder in einer Mischung aus Wasser und einem mit Wasser mischbaren, organischen Lösungsmittel löst oder

a_2) ein oder mehrere Phytosterole in einem mit Wasser nicht mischbaren, organischen Lösungsmittel löst und

b) die nach a_1) oder a_2) erhaltene Lösung mit einer wäßrigen molekulardispersen oder kolloiddispersen Lösung eines Schutzkolloids mischt, wobei die hydrophobe Phase des Phytosterols als disperse Phase entsteht, und

c) die gebildete Dispersion für die Herstellung eines Trockenpulvers von dem Lösungsmittel und dem Wasser befreit und, gegebenenfalls in Gegenwart eines Überzugsmaterials, trocknet.

[0027] Je nach Art der verwendeten Lösungsmittel kann es sich bei der dispersen Phase im Schritt b) um feste Nanopartikel (Suspension) oder um Nanotröpfchen (Emulsion) handeln.

[0028] Die in der Stufe a_2) verwendeten wassermischbaren Lösungsmittel sind vor allem wassermischbare, thermisch stabile, flüchtige, nur Kohlenstoff, Wasserstoff und Sauerstoff enthaltenene Lösungsmittel wie Alkohole, Ether, Ester, Ketone und Acetate zu nennen.

[0029] Zweckmäßig verwendet man solche Lösungsmittel, die mindestens zu 10 % wassermischbar sind, einen Siedepunkt unter 200°C aufweisen und/oder weniger als 10 Kohlenstoffe haben. Besonders bevorzugt werden Methanol, Ethanol, n-Propanol, Isopropanol, 1,2-Butandiol-1-methylether, 1,2-Propandiol-1-n-propylether, Tetrahydrofuran und/oder Aceton, ganz besonders bevorzugt n-Pro-

panol, Isopropanol und/oder Aceton verwendet.

[0030] Der Begriff "ein mit Wasser nicht mischbares organisches Lösungsmittel" steht im Sinne der vorliegenden Erfindung für ein organisches Lösungsmittel mit einer Wasserlöslichkeit bei Normaldruck von weniger als 10%. Als mögliche Lösungsmittel kommen dabei u.a. halogenierte aliphatische Kohlenwasserstoffe, wie z.B. Methylenchlorid, Chloroform und Tetrachlorkohlenstoff, Carbonsäureester wie Dimethylcarbonat, Diethylcarbonat, Propylencarbonat, Ethylformiat, Methyl-, Ethyl- oder Isopropylacetat sowie Ether wie Methyl-tert. butylether in Frage. Bevorzugte, mit Wasser nicht mischbare organische Lösungsmittel sind die folgenden Verbindungen aus der Gruppe, bestehend aus Dimethylcarbonat, Propylencarbonat, Ethylformiat, Ethylacetat, Isopropylacetat und Methyl-tert. butylether.

[0031] Als Schutzkolloide werden im Verfahrensschritt b) die bereits eingangs genannten Verbindungen eingesetzt.

[0032] Unter Umständen kann es auch vorteilhaft sein, zusätzlich zu der Lösungsmittel-Phase ein physiologisch zugelassenes Öl wie beispielsweise Sesamöl, Maiskeimöl, Baumwollsaatöl, Sojabohnenöl oder Erdnußöl sowie Ester mittelkettiger pflanzlicher Fettsäuren in einer Konzentration von 0 bis 500 Gew.-%, vorzugsweise 10 bis 300 Gew.-%, besonders bevorzugt 20 bis 100 Gew.-%, bezogen auf das/die Phytosterol(e), zu geben, das dann gemeinsam mit den Wirkstoffen und den genannten Zusatzstoffen beim Mischen mit der wässrigen Phase extrem feinteilig ausgefällt wird.

[0033] Eine bevorzugte Ausführungsform des erfindungsgemäßen Verfahrens ist dadurch gekennzeichnet, dass man

a) ein oder mehrere Phytosterole in einem mit Wasser mischbaren, organischen Lösungsmittel oder einer Mischung aus Wasser und einem mit Wasser mischbaren, organischen Lösungsmittel bei Temperaturen im Bereich von 50°C bis 240°C, bevorzugt im Bereich von 100°C bis 200°C, besonders bevorzugt von 140°C bis 180°C löst,

b) die erhaltene Lösung mit einer wäßrigen molekulardispersen oder kolloiddispersen Lösung eines Schutzkolloids, ausgewählt aus der Gruppe, bestehend aus Pektin, Kasein, Natrium-Kaseinat, Gummi Arabicum, modifizierte Stärke und Fischgelatine mischt, wobei sich eine Mischungstemperatur von etwa 35°C bis 80°C einstellt und

c) die gebildete Dispersion in ein Trockenpulver überführt.

[0034] Ganz besonders bevorzugt handelt es sich hierbei um ein Verfahren zur Herstellung von Trockenpulvern einer Mischung aus Stigmasterol, Campesterol und ß-Sitosterol.

[0035] Da die Einwirkung hoher Temperaturen u. U. den gewünschten Gehalt an Phytosterolen herabsetzen kann, löst man das/die Phytosterol(e) möglichst rasch, beispielsweise im Sekundenbereich, z.B. in 0,1 bis 10 Sekunden, besonders bevorzugt in weniger als 1 Sekunde. Zur raschen Herstellung der molekulardispersen Lösung 5 kann die Anwendung von erhöhtem Druck, z.B. im Bereich von 20 bar bis 80 bar, vorzugsweise 30 bis 60 bar, vorteilhaft sein.

[0036] Die so erhaltene molekulardisperse Lösung versetzt man anschließend direkt mit der gegebenenfalls gekühlten wäßrigen molekulardispersen oder kolloiddispersen Lösung des Schutzkolloids in der Weise, daß sich eine Mischungstemperatur von etwa 35°C bis 80°C einstellt.

[0037] Dabei wird die Lösungsmittelkomponente in die wäßrige Phase überführt und die hydrophobe Phase des/der Phytosterole/Phytosterole entsteht als disperse Phase.

[0038] Die mittlere Teilchengröße der nanopartikulären Teilchen in der wäßrigen Dispersion liegt je nach Art der Formulierungsmethode im Bereich von 0,01 bis 100 µm, bevorzugt im Bereich von 0,01 bis 10 µm, besonders bevorzugt im Bereich von 0,01 bis 2 µm, ganz besonders bevorzugt im Bereich von 0,05 bis 1 µm.

[0039] Hinsichtlich einer näheren Verfahrens- und Apparatebeschreibung zur oben genannten Dispergierung wird an dieser Stelle auf EP-B-0 065 193 Bezug genommen.

[0040] Die Überführung in ein Trockenpulver kann dabei u.a. durch Sprühtrocknung, Sprühkühlung, Gefriertrocknung oder Trocknung im Wirbelbett, gegebenenfalls auch in Gegenwart eines Überzugsmaterials erfolgen. Als Überzugsmittel eignen sich u.a. Maisstärke, Kieselsäure oder auch Tricalciumphosphat.

[0041] Gegenstand der Erfindung ist auch ein Verfahren zur Herstellung der oben genannten pulverförmigen Phytosterol-Formulierungen, dadurch gekennzeichnet, dass man mindestens ein Phytosterol in einem wäßrigen Medium in Gegenwart eines Schutzkolloids mahlt und die so erhaltene Phytosterol Suspension für die Herstellung eines Trockenpulvers trocknet.

[0042] Die Mahlung kann dabei in an sich bekannter Weise z.B. mit einer Kugelmühle erfolgen. Dabei wird je nach verwendetem Mühlentyp so lange gemahlen, bis die Teilchen eine mittlere Partikelgröße von 0,01 bis 100 µm, bevorzugt 0,2 bis 50 µm, besonders bevorzugt 0,2 bis 20 µm, ganz besonders bevorzugt 0,2 bis 5 µm, insbesondere 0,2 bis 0,8 µm aufweisen.

[0043] Nähere Einzelheiten zur Mahlung und den dafür verwendeten Apparaturen finden sich u.a. in Ullmann's Encyclopedia of Industrial Chemistry, Sixth Edition, 1999, Electronic Release, Size Reduction, Kapitel 3.6.: Wet Grinding sowie in EP-A-0 498 824.

[0044] Eine weitere Variante des oben genannten Mahlverfahrens ist dadurch gekennzeichnet, dass man die Phytosterol Suspension nach dem Mahlen auf eine ausreichend hohe Temperatur erhitzt, um ein vollständiges oder teilweises Schmelzen der Phytosterole zu bewirken, und man diese Schmelze vor der Überführung in ein Trockenpulver wieder abkühlt. Be-

vorzugt wird dabei die Phytosterol Suspension nach dem Mahlen über einen Zeitraum von 0,05 bis 200 Sekunden, bevorzugt 0,2 bis 100 Sekunden auf eine Temperatur von 150 bis 200°C gehalten und vor der Überführung in ein Trockenpulver auf eine Temperatur von zwischen 20 und 80°C abgekühlt.

[0045] Je nach Trocknungsmethode weisen die Phytosterol-haltigen Trockenpulver eine mittlere Partikelgröße von 100 bis 1000 µm bevorzugt von 200 bis 800 µm, besonders bevorzugt von 250 bis 600 µm auf. Diese Pulver stellen Agglomerate (Sekundärteilchen) der bereits eingangs mit einer mittleren Teilchengröße im Bereich von 0,01 bis 100 µm beschriebenen Primärteilchen dar.

[0046] Die Bestimmung der Partikelgröße, sowohl der Primär- als auch der Sekundärteilchen, erfolgt dabei mit Hilfe bekannter Meßmethoden u.a. über Fraunhofer Beugung sowie bei Partikeln kleiner 5 µm mit Hilfe dynamischer Lichtstreuung.

[0047] Die erfindungsgemäßen Trockenpulver zeichnen sich u.a. dadurch aus, daß sie sich in wäßrigen Systemen unter Erzielung einer gleichmäßigen Feinverteilung des Wirkstoffes im Korngrößenbereich von 0,01 bis 1 µm problemlos wieder redispergieren lassen.

[0048] Die erfindungsgemäßen Phytosterol-Formulierungen eignen sich u.a. als Zusatzstoff für Lebensmittelzubereitungen und Tierfuttermittel, als Mittel für die Herstellung pharmazeutischer und kosmetischer Zubereitungen sowie für die Herstellung von Nahrungsergänzungspräparaten im Human- und Tierbereich.

[0049] Ein typisches Einsatzgebiet im Lebensmittelbereich ist beispielsweise die Verwendung in Getränken, Milchprodukten wie Käse, Joghurt, Milchmixgetränke oder Milchspeiseeis sowie in Salatdressings, Saucen und Mayonnaisen aber auch in Wurstwaren und in Süßwaren.

[0050] Bevorzugt lassen sich die Suspensionen als Futtermittelzusatz in der Tierernährung einsetzen, insbesondere zum Auftragen bzw. Aufsprühen auf Futtermittelpellets.

[0051] Die Anwendung als Futtermittelzusatzstoff erfolgt insbesondere in Form flüssiger Zubereitungen, in denen die erfindungsgemäßen pulverförmigen Phytosterol-Formulierungen in einem Öl dispergiert vorliegen.

[0052] Als Öle kommen dabei in der Regel alle physiologisch unbedenklichen Öle – sowohl pflanzlichen als auch tierischen Ursprungs – in Frage, insbesondere solche Öle, die bei 20°C flüssig sind bzw. die in der Suspension bei 20°C allein oder zusammen mit anderen Ölen die flüssige Phase bilden. Bevorzugt zu nennen sind in diesem Zusammenhang Sonnenblumenöl, Palmöl, Sesamöl, Maiskeimöl, Baumwollsaatöl, Sojabohnenöl oder Erdnußöl, Ester mittelkettiger Triglyceride sowie außerdem Fischöle wie beispielsweise Makrelen-, Sprotten- oder Lachsöl. Für die Tierernährung besonders bevorzugt sind Fischöle, Maiskeimöl, Sonnenblumenöl und Erdnußöl.

[0053] Diese flüssigen Zubereitungen können beispielsweise durch direktes Aufsprühen auf Tierfutterpellets als sogenannte "post-pelleting-application" verabreicht werden.

[0054] Eine bevorzugte Ausführungsform des Sprühverfahrens besteht darin, daß man beispielsweise die Futtermittelpellets unter vermindertem Druck mit der öligen Suspension beladt.

[0055] Beispiele hierfür finden sich u.a. in GB-A-2 232 573 sowie in EP-A-0 656 883.

[0056] Typische Einsatzgebiete im Lebensmittelbereich sind beispielsweise die Vitaminierung von Getränken, Milchprodukten wie Joghurt, Milchmixgetränken oder Milchspeiseeis sowie von Puddingpulvern, Eiprodukten, Backmischungen und Süßwaren.

[0057] Im Kosmetikbereich können die öligen Suspensionen beispielsweise für Vitamin-haltige Körperpflegemittel beispielsweise in Form einer Creme, einer Lotion, als Lippenstifte oder Make-up verwendet werden.

[0058] Im Kosmetikbereich können die erfindungsgemäßen Phytosterol-Formulierungen beispielsweise als Emollient oder auch als Wirkstoff in Hautpflegemittel verwendet werden.

[0059] Gegenstand der Erfindung sind auch Nahrungsergänzungsmittel, Tierfuttermittel, Lebensmittel sowie pharmazeutische und kosmetische Zubereitungen, enthaltend die oben beschriebenen Phytosterol-Formulierungen.

[0060] Unter Nahrungsergänzungspräparate sowie pharmazeutische Zubereitungen, die die erfindungsgemäße Phytosterol-Formulierung enthalten, sind u.a. Tabletten, Dragees sowie Hart- und Weichgelatinekapseln zu verstehen.

[0061] Als Lebensmittel sind beispielsweise Getränke, Milchprodukte wie Käse, Joghurt, Milchmixgetränke oder Milchspeiseeis sowie Salatdressings, Saucen oder Mayonnaisen Süßwaren und Wurstwaren gemeint, die die oben beschriebenen Phytosterol-Formulierungen enthalten.

[0062] Kosmetische Zubereitungen, die die erfindungsgemäßen Phytosterol-Formulierungen enthalten können, sind beispielsweise topisch anwendbare Zubereitungen, insbesondere Hautpflegemittel und dekorative Körperpflegemittel wie Lippenstifte, Gesichts-Make-up in Form einer Creme, einer Lotion, eines Puders oder auch als Rouge.

[0063] Die pharmazeutischen Zubereitungen eignen sich zur Prophylaxe oder Therapie eines zu hohen Cholesterinspiegels.

[0064] In den nachfolgenden Beispielen wird die Herstellung der erfindungsgemäßen Phytosterol-Formulierungen näher erläutert. Einzelheiten zu der in den Beispielen verwendeten Apparatur finden sich in EP-B-0 065 193.

Beispiel 1

Phytosterol Trockenpulver mit Natrium-Kaseinat

[0065] In einer Vorlage wurden 21 g Phytosterol (Fa. ADM, USA) und 2,1 g Ascorbylpalmitat in 360 g Aceton bei Raumtemperatur gelöst. In einer zweiten Vorlage wurden 35 g Na-Kaseinat und 35 g Saccharose in 4000 g vollentsalztem Wasser bei 70°C gelöst. Die Losemittelphase, die auf 86,8°C eingestellt war, wurde anschließend mit einer Pumprate von 9,92 kg/h mit der wässrigen Phase bei Raumtemperatur mit einer Pumprate von 30,3 kg/h kontinuierlich vermischt. Die so entstandene Wirkstoffdispersion wurde an einem Rotationsverdampfer bei 65°C und einem Druck von 200 mbar von Aceton befreit und auf einen Feststoffgehalt von 11,5 Gew.-% aufkonzentriert. Die dabei entstandenen Wirkstoffteilchen wiesen eine Teilchengröße von 203 nm auf.

[0066] Anschließend wurde diese Dispersion auf einem Laborsprühturm sprühgetrocknet. Der Phytosterolgehalt in dem so erhaltenen Trockenpulver betrug 25 Gew.-%. Das Trockenpulver ist in Wasser dispergierbar und nach Redispergierung ergab sich eine Teilchengröße von 1,08 µm.

Beispiel 2

Phytosterol Trockenpulver mit modifizierter Stärke

[0067] In einer Vorlage wurden 21 g Phytosterol (Fa. ADM, USA) und 2,1 g Ascorbylpalmitat in 360 g Aceton bei Raumtemperatur gelöst. In einer zweiten Vorlage wurden 35 g modifizierte Stärke (Emcap 12633, Fa. Cerestar, Krefeld) und 35 g Saccharose in 4000 g vollentsalztem Wasser bei 70°C gelöst. Die Lösemittelphase, die auf 94,9°C eingestellt war, wurde anschließend mit einer Pumprate von 2,61 kg/h mit der wässrigen Phase bei Raumtemperatur mit einer Pumprate von 30,0 kg/h kontinuierlich vermischt. Die so entstandene Wirkstoffdispersion wurde an einem Rotationsverdampfer bei 65°C und einem Druck von 200 mbar von Aceton befreit und auf einen Feststoffgehalt von 9,1 Gew.-% aufkonzentriert. Die dabei entstandenen Wirkstoffteilchen wiesen eine Teilchengröße von 264 nm auf.

[0068] Anschließend wurde diese Dispersion auf einem Laborsprühturm sprühgetrocknet. Der Phytosterolgehalt in dem so erhaltenen Trockenpulver betrug 20,7 Gew.-%. Das Trockenpulver ist in Wasser dispergierbar und nach Redispergierung ergab sich eine Teilchengröße von 2,3 µm.

Beispiel 3

Phytosterol Trockenpulver mit modifizierter Stärke

[0069] 40 g Phytosterol (Fa. ADM, USA), 6 g Ascorbylpalmitat und 40 g modifizierte Stärke (Capsul MKH, Fa. National Starch, Hamburg) wurden bei Raumtemperatur in 400 g vollentsalztem Wasser suspendiert. Der pH-Wert wurde anschließend mit 1 M NaOH auf pH 7,1 eingestellt. Diese Suspension wurde dann zusammen mit 2000 g Keramikkugeln (Zirkoniumoxid, Toray) des Durchmessers 1 mm in eine 1000 ml Glasflasche gegeben. Die Suspension wurde dann in dieser Glasflasche 8 Stunden auf einem Dispergiergerät (Red Devil) dispergiert. Die Wirkstoffteilchen hatten danach eine Größe von 585 nm.

[0070] Nach Abtrennung der Mahlkörper wurde 344 g Dispersion erhalten. In dieser wurden 28,3 g Saccharose gelöst. Anschließend wurde diese Dispersion auf einem Laborsprühturm sprühgetrocknet. Der Phytosterolgehalt in dem so erhaltenen Trockenpulver betrug 19,2 Gew.-%. Das Trockenpulver ist in Wasser dispergierbar und nach Redispergierung ergab sich eine Teilchengröße von 1,2 µm.

Patentansprüche

1. Pulverförmige Phytosterol-Formulierungen, enthaltend mindestens ein Phytosterol mit einer mittleren Teilchengröße von 0,01 bis 100 µm.

2. Phytosterol-Formulierungen nach Anspruch 1, dadurch gekennzeichnet, dass mindestens ein Phytosterol in teilamorpher Form vorliegt.

3. Phytosterol-Formulierungen nach einem der Ansprüche 1 oder 2, dadurch gekennzeichnet, dass das Phytosterol in einer Schutzkolloid-Matrix eingebettet ist.

4. Phytosterol-Formulierungen nach einem der Ansprüche 1 bis 3, enthaltend 0,1 bis 80 Gew.-% eines oder mehrerer Phytosterole, wobei sich die Gew.-Prozentangaben auf die Trockenmasse des Pulvers beziehen.

5. Phytosterol-Formulierungen nach Anspruch 4, enthaltend 5 bis 70 Gew.-% eines oder mehrerer Schutzkolloide.

6. Phytosterol-Formulierungen nach einem der Ansprüche 4 oder 5, enthaltend zusätzlich 0,1 bis 70 Gew.-% eines oder mehrerer Weichmacher.

7. Phytosterol-Formulierungen nach einem der Ansprüche 4 bis 6, enthaltend zusätzlich 0,01 bis 70 Gew.-% eines oder mehrerer Emulgatoren.

8. Phytosterol-Formulierungen nach einem der Ansprüche 4 bis 7, enthaltend zusätzlich 0,01 bis 50 Gew.-% eines oder mehrerer Antioxidantien und/oder Konservierungsmittel.

9. Phytosterol-Formulierungen nach einem der Ansprüche 1 bis 8, dadurch gekennzeichnet, dass sie wasserdispergierbar sind.

10. Verfahren zur Herstellung von pulverförmigen Phytosterol-Formulierungen, definiert gemäß Anspruch 1, dadurch gekennzeichnet, dass man
a_1) ein oder mehrere Phytosterole in einem mit Wasser mischbaren, organischen Lösungsmittel oder in einer Mischung aus Wasser und einem mit Wasser mischbaren, organischen Lösungsmittel löst oder
a_2) ein oder mehrere Phytosterole in einem mit Wasser nicht mischbaren, organischen Lösungsmittel löst und
b) die nach a1) oder a2) erhaltene Lösung mit einer wäßrigen molekulardispersen oder kolloiddispersen Lösung eines Schutzkolloids mischt, wobei die hydrophobe Phase des Phytosterols als disperse Phase entsteht, und
c) die gebildete Dispersion für die Herstellung eines Trockenpulvers von dem Lösungsmittel und dem Wasser befreit und, gegebenenfalls in Gegenwart eines Überzugsmaterials, trocknet.

11. Verfahren nach Anspruch 10, dadurch gekennzeichnet, dass man
a) ein oder mehrere Phytosterole in einem mit Wasser mischbaren, organischen Lösungsmittel oder einer Mischung aus Wasser und einem mit Wasser mischbaren, organischen Lösungsmittel bei Temperaturen im Bereich von 50°C bis 240°C löst,
b) die erhaltene Lösung mit einer wäßrigen molekulardispersen oder kolloiddispersen Lösung eines Schutzkolloids, ausgewählt aus der Gruppe, bestehend aus Pektin, Kasein, Kaseinat, Gummi Arabicum, modifizierte Stärke und Fischgelatine mischt, wobei sich eine Mischungstemperatur von etwa 35°C bis 80°C einstellt und
c) die gebildete Dispersion in ein Trockenpulver überführt.

12. Verfahren zur Herstellung von pulverförmigen Phytosterol-Formulierungen, definiert gemäß Anspruch 1, dadurch gekennzeichnet, dass man mindestens ein Phytosterol in einem wäßrigen Medium in Gegenwart eines Schutzkolloids mahlt und die so erhaltene Phytosterol Suspension für die Herstellung eines Trockenpulvers trocknet.

13. Verfahren nach Anspruch 12, dadurch gekennzeichnet, dass man die Phytosterol Suspension nach dem Mahlen auf eine ausreichend hohe Temperatur erhitzt, um ein vollständiges oder teilweises Schmelzen der Phytosterole zu bewirken, und man diese Schmelze vor der Überführung in ein Trockenpulver wieder abkühlt.

14. Verfahren nach Anspruch 13, dadurch gekennzeichnet, dass die Phytosterol Suspension nach dem Mahlen über einen Zeitraum von 0,05 bis 200 Sekunden auf eine Temperatur von 150 bis 200°C gehalten wird und vor der Überführung in ein Trockenpulver auf eine Temperatur von zwischen 20 und 80°C abgekühlt wird.

15. Verwendung der Phytosterol-Formulierungen, definiert gemäß Anspruch 1 zur Herstellung von Nahrungsergänzungsmitteln sowie als Zusatz zu Lebensmitteln, Tierfuttermitteln, pharmazeutischen und kosmetischen Zubereitungen.

16. Nahrungsergänzungsmittel, Tierfuttermittel, Lebensmittel sowie pharmazeutische und kosmetische Zubereitungen, enthaltend Phytosterol-Formulierungen, definiert gemäß Anspruch 1

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(54) Particulate creamer comprising fat and food compositions comprising said creamer

(57) Particulate compositions comprising 10-90%wt of triglycerides of fatty acids, which particulates are at least partly covered or encapsulated by 10-90%wt of an encapsulating or covering material, wherein the amount of H3 (triglyceride of 3 saturated fatty acids of 16 or more carbon atoms) and H2U (triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 cis-unsaturated fatty acid) taken together is at least 55%wt based on the total amount of triglycerides, wherein the compositions are low in triglycerides of transunsaturated fatty acids, for use as for exemple creamer and/or whitener. The invention further relates to food products containing such creamer.

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Description

Field of the invention

[0001] The present invention relates to particulate compositions suitable for use as creamer and/or whitener, and in particular comprising triglycerides of fatty acids, wherein the composition is low in triglycerides of trans-unsaturated fatty acids. The invention further relates to food products containing such creamer.

Background of the invention

[0002] Food compositions (and in particular savoury food compositions) contain in many cases fat. This is especially the case for particulate and/or pasty compositions like preparations for instant cream-style soups and sauces, which to a large extent consist of fat, starch or a starchy matter, and salt and flavourings. Particulate in this context is to be understood as powder, flakes, cubes, pellets etcetera.

[0003] (Mixtures for) cream-style soups and sauces as above referred, but also other products such as instant dishes like pasta with a sauce often contain an ingredient which is referred to as a creamer, and/or creamer/whitener, and/or creamer/thickener, and/or non-dairy creamer. These are usually fat blends that can provide a creamy taste and mouth-feel, improved body and/or viscosity and/or a whitening effect. Such creamers can also be in the form of e.g. tablets as a non-dairy cream alternative. To be suitable in these applications the fat blends must have the appropriate physical properties in terms of melting behaviour, crystallisation behaviour, brittleness, organoleptic properties, taste, as well as physical and chemical stability. In order to increase stability, shelf life and solubility, the fat blends are commonly encapsulated or (partly) coated with another material, e.g. hydrophilic film forming materials. In such encapsulates or partly coated fats, the individual fat blend particles as well as clusters of fat blend particles are at least partially covered and/or surrounded by the encapsulation material. The fat blends should therefore also be suitable for being submitted to encapsulation and drying processes in order to form free flowing and highly dispersible products.

[0004] The fats in savoury compositions described above usually comprise a considerable amount triglycerides of fatty acids (hereinafter for short: triglycerides). Fats are usually mixtures of various triglycerides. The type of fat or fat blend used for a given purpose is determined (next to availability and price) by e.g. the properties the fat has and how it performs in a given product, and in the manufacture of such product. The fat should perform well on e.g. taste, melting in the mouth, taste keepability, but also on ability to be processed into a suitable product as well as performance in the packed product, e.g. keepability (in particular fat staining for cubes packaged in cardboard).

[0005] The triglycerides (which form part or all of the fat) are usually obtained from vegetable sources and may have been subjected to various treatments, such as fractionation (dry or wet), purification, hardening, interesterification, blending etcetera, to give the fat the desired product properties. Hardening unsaturated fat or triglycerides to saturated or partially unsaturated fat or triglycerides is in particular a tool used to obtain the desired melting behaviour. In this way, oils or soft fats can be turned into fats showing more suitable properties for solid or dry formulations.

[0006] The hardening process may lead to formation of a certain amount of so-called trans-unsaturated fatty acids (and/or triglycerides containing such trans-unsaturated fatty acids), in short TFA's. For various reasons it may be desired to reduce or eliminate the amount of trans-unsaturated fatty acids (and triglycerides thereof) in products. For spreads (margarines and the like) a wide range of possible alternative fats and triglycerides are proposed, as is disclosed in e.g. WO 97/16978 and WO 98/39956.

[0007] The triglycerides mentioned in such applications frequently contain lauric acid (C12 saturated fatty acid). It has been found that when one wishes to find an alternative for the trans-unsaturated fatty acids (and fats containing them) in savoury applications (in which the creamers are often used) lauric acid (and its triglycerides) is undesired. Lauric acid and triglycerides containing lauric acid may show a range of desirable properties, in particular melting behaviour, but in a savoury application triglycerides of lauric acid were found to lead to a (soapy) off-flavour, especially after prolonged storage.

[0008] Hence, there is a desire for creamer, and/or creamer/whitener, and/or creamer/thickener, and/or non-dairy creamer alternative and also (savoury) food compositions such as (mixtures for) cream-style soups and sauces, (instant) food compositions, meal makers and others that contain such creamer, or creamer/whitener, and/or creamer/thickener, and/or non-dairy creamer alternative, wherein the creamer, and/or creamer/whitener, and/or creamer/thickener, and/or non-dairy creamer alternative which contain triglyceride fats which are low in trans-unsaturated fatty acids (e.g. below 5% of the total fats present, preferably less than 2%). Still such creamer, and/or creamer/whitener, and/or creamer/thickener, and/or non-dairy creamer alternative should be not too difficult to manufacture in comparison to the conventional products, and should still perform well in a (savoury) food composition comprising carbohydrates, in particular concerning processability, fat staining, crystallisation, mouthfeel, and other characteristics as mentioned above.

[0009] Preferably, such product should also be low in lauric acid or triglycerides thereof (e.g. below 10% of the total

fats present, preferably less than 3%, most preferably less than 0.5% wt of the total fats present). Also, the alternative fats should combine well with the coating /encapsulation material. Suitable encapsulation material for the creamers etcetera according to the invention are edible proteins such as for example milk proteins, hydrolysed proteins, edible carbohydrates, such as for example starch or modified starches as well as sugars or sugar derivatives, dextrines or maltodextrines etcetera. The creamers in the form of encapsulated or covered fat usually contain 20-80% wt of encapsulation material, based on the total creamer. The manufacturing process usually involve a drying step, such as e.g. spray-dried but other drying processes such as for example heat drying (including vacuum freeze drying), air drying etc can also be employed.

[0010] Triglyceride fats can be grouped according to the fatty acids of which they consist. Such groups can be identified by a letter, and herein:

H means saturated fatty acid of 16 carbon atoms or longer (C16+, e.g. up to C24)
U means unsaturated fatty acids in cis conformation (any chain length)
E means unsaturated fatty acids in trans conformation (any chain length)
M means saturated fatty acids of 10-14 carbon atoms (C10-C14)

As the present application is about triglycerides of such fatty acids, the fatty acid composition of the triglycerides is given by for example:

H3 (meaning a triglyceride of 3 saturated fatty acids of 16 or more carbon atoms)
H2E (meaning a triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 trans-unsaturated fatty acid)
H2M (meaning a triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 saturated fatty acid of 10-14 carbon atoms)
H2U (meaning a triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 cis-unsaturated fatty acid)
HE2 (meaning a triglyceride of 1 saturated fatty acid of 16 or more carbon atoms and 2 trans-unsaturated fatty acids), and so on for other 3 letter codes.

[0011] Fat compositions can thus be characterised in containing certain weight percentages (based on the total amount of triglycerides) of triglycerides of the above codes.

[0012] Although it is mentioned for E and U that they may have any length, it is to be understood that this relates to fatty acids of approx. 8-24 carbon atoms, and more usually 16-20 carbon atoms.

Summary of the invention

[0013] It has now been found that the objectives as given above may be met (at least in part) by particulates comprising 10-90% wt (preferably 15-80% wt) of triglycerides of fatty acids, which particulates are at least partly covered or encapsulated by 10-90% wt (preferably 20-85%) of an encapsulating or covering material, wherein the amount of H3 (triglyceride of 3 saturated fatty acids of 16 or more carbon atoms) and H2U (triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and one cis-unsaturated fatty acid) taken together in the creamer particulates is at least 55% wt based on the total amount of triglycerides, preferably at least 65% % wt based on the total amount of triglycerides. In other words: H3 + H2U > 55%, preferably > 65% wt, based on total triglycerides.

[0014] This means that of the total amount of triglycerides present in the particulates at least 55% wt (preferably at least 65% wt) are triglycerides of fully saturated C16 and longer chains (e.g. C16, C18, C20, C22 and C24) and/or triglycerides containing one cis-unsaturated fatty acid of any chain length and two saturated fatty acids of 16 or more carbon atoms. In connection to this, it is believed that the creamers, whiteners, non-dairy cream alternatives, etcetera as currently on the market in particulate form contain about 30-50% of such H3 + H2U triglycerides as part of their fats.

[0015] The preparations according to the invention are often dry preparations. However, such compositions still may contain a considerable amount of water, e.g. as a result of an incomplete dehydration process or as a result from water naturally present in the constituents, such as moisture in flour. The amount of moisture present in the compositions according to the invention is below 30% wt (based on the total composition), preferably less than 20% wt, more preferably less than 10%wt.

[0016] As is mentioned before, the fat blends are encapsulated by or (partly) coated with another material, e.g. hydrophilic film forming materials. In such encapsulates or partly coated fats, the individual fat blend particles as well as clusters of fat blend particles are at least partially covered and/or surrounded by the encapsulation material. Such covering or encapsulation material preferably comprises a protein and/or a carbohydrate. Examples of such proteins protein are dairy protein, hydrolysed protein, gelatin, soy protein, or mixtures thereof. Examples of such carbohydrate are maltodextrin, a sugar, a sugar derivative, starch, chemically modified starch, physically modified starch, xanthan, guar, locust bean gum, alginate, carrageenan, polydextrose, or mixtures thereof.

[0017] Following the invention, it is now possible to manufacture particulates, suitable as creamer and/or whitener and/or non-dairy cream alternative in food compositions, comprising 10-90% wt (preferably 15-80% wt) of triglycerides of fatty acids, which particulates are at least partly covered or encapsulated by 10-90% wt (preferably 20-85%) of an encapsulating or covering material, wherein the triglycerides are selected such that they contain less than 5%, preferably less than 2% wt of trans-unsaturated fatty acids, and preferably having less then 10% wt (more preferably less than 3%, most preferably less then 0.5%) of lauric acid, and wherein the fats still have appropriate melting and crystallisation behaviour.

[0018] Hence, it is now possible to manufacture e.g. creamer and/or whitener and/or non-dairy cream alternative-type products which contain a large proportion of vegetable fats, which composition is substantially free from trans-fatty acids or triglycerides thereof. Thus, the invention further relates to a creamer and/or whitener and/or non-dairy cream alternative-type product comprising 10-90% wt (preferably 15-80% wt) of triglycerides of fatty acids, which particulates are at least partly covered or encapsulated by 10-90% wt (preferably 20-85%) of an encapsulating or covering material wherein at least 50% of the fats is of vegetable origin, and which composition is substantially free from trans-unsaturated fatty acids or triglycerides thereof. Preferably, the compositions according to the invention are substantially free from animal fat.

[0019] The invention also relates to (savoury) food compositions comprising the particulates according to the invention.

Detailed description of the invention

[0020] In the particulates according to the invention it is preferred that the amount of H3 (triglyceride of 3 saturated fatty acids of 16 or more carbon atoms) is at least 15% wt based on the total amount of triglycerides in the particulates, preferably at least 20%. Likewise, it is preferred that the amount of H2U (triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 cis-unsaturated fatty acid) taken together is at least 40% wt based on the total amount of triglycerides in the particulates.

[0021] Apart from the amounts of H3 and H2U it can be preferred to use fats in such particulates in a particular ratio. In this case, the ratio H3 : H2U is preferably between 0.5 and 1.2.

[0022] Regarding the basic fatty acid composition, it is preferred that the amount of H (i.e. saturated fatty acids of 16 or more carbon atoms) is between 60 and 75% wt based on total amount of fatty acids. Normally, only fatty acids are used with even number of carbon atoms. Similarly, it is preferred that the amount of U (cis-unsaturated fatty acids of any suitable chain length) is between 20 and 45% wt based on total amount of fatty acids.

[0023] In the particulates according to the invention the amount of palmitic fatty acid (C16:0) in the triglycerides is preferably between 40 and 60% wt based on the total amount of fatty acids.

[0024] The invention thus also relates to (savoury) food compositions comprising the particulates as set out above, such as sauce and soup concentrates. Such (savoury) food compositions according to the invention can be any physical format, but the invention is most suitable for savoury compositions that are in the form of pasty or particulate matter. Particulate matter is herein to be understood to comprise e.g. flakes, powder, cubes, pellets, tablets. In the case of cubes, pellets, tablets it may be needed to use a technique such as agglomerating or pressing the particulates according to the invention to obtain such shapes. The (savoury) food compositions as set out above usually contain additional material, such as 2-50% wt salt, 0-30% wt MSG, 0-20% wt herbs and/or spices, 0-30% wt vegetable particulates, 0-30% wt starch-based thickener and further comprising 0.1-30% wt of the particulates according to the invention.

[0025] The savoury food compositions according to the invention will may further comprise (e.g. in an amount of 0.1-50% wt) one or more of the following ingredients: herbs and/or spices, tomato powder, vegetable pieces, monosodium glutamate and other components.

EXAMPLES

Example 1: manufacture of five different creamers

[0026] Five creamers were prepared having the net composition as given in the table below, by starting with a fat phase and an aqueous phase.

Creamer ingredients

Ingredients	Carboydrates*	*Fat	*Proteines	*Others	Total
Product	(%)	(%)	(%)	(%)	(%)
Creamer a	69	15	14	2	100
Creamer b	11,5	75	10	3,5	100
Creamer c	15	78	7	0	100
Creamer d	20,5	65	14	0,5	100
Creamer e	44	50	6	0	100

***Carbohydrates**
wheat flour
Maltodextrin
Glucose syrup
***Proteines**
Milkproteinss
***Others**
Phosphate
citrate
***Fat**
fat blends

fat phase

[0027] Fat blends A-D were prepared from various known fats and fats specifically produced according to the top half of the table below. In the second half of that table the fatty acid composition is given in accordance with the difinitions as herein defined, as is the ratio symmetric: assymmetric triglycerides for H2U.

[0028] 2 Kg fat blend was prepared by mixing 1,2kg POs and 0 8 kg PO (blend A in the table below) and heated up 75°C in a blending vessel under nitrogen atmosphere for 10 min.

Other fat blends which can be used are B-D, with good properties. PO is palm oil. POs is a dry-fractionated palm stearin with a melting point of approx. 53°C. PO58 is fully hardened palm oil. Fatblend "comp" is a comparative, from the prior art, obtained by hardenening palm oil to a melting point of approx. 44°C.

	comp	A	B	C	D
PO		40	30	40	20
POs		60	70	50	80
PO58				10	
PO44	100				
H3	12	26	27	31	29
H2E	29	0	0	0	0
H2M	1	1	1	2	1
H2U	21	46	45	42	44

(continued)

	comp	A	B	C	D
HE2	10	0	0	0	0
H3/H2U	0.55	0.54	0.60	0.74	0.67
H	53	61	63	64	64
E	24	0	0	0	0
U	23	39	37	36	36
Palmitic	45	54	56	53	57
H3+H2U	33	71	72	72	73
Note: the numbers of all the fats given does not add up to 100%, as some minor amounts of other fats are also present.					

aqueous phase

[0029]

7 kg of an aqueous phase was prepared mixing:
5 kg water
1.3 kg fat blend/fat phase as above
0.22 kg maltodextrin
0.28 kg milkprotein
0.19 kg lactose
0.01 kg citrate

[0030] in a mixing tank with a Ultraturrax for 5 min. at 55°C and then homogenized in a homogenizer (Schroeder) one stage 200 bar. The resulting suspension then was spray dried in a multi stage spray dryer (Niro).
The inlet temperature was about 165 °C; the outlet temperature about 82°C.
The dry particulate creamer was agglomerated for 5 minutes in an agglomeration process step

(Glatt Agglomator)	
Temperature:	
Inlet temperature	80 °C
Outlet temperature	50 °C

[0031] The spray dried and agglomerated creamer was stored under cool conditions below 20°C and used in the formulations according to the examples below.

Example 2: Saffron cream soup

[0032] A dry soup mix for a saffron cream soup was made by mixing:

Creamer as in example 1-c	32,94%
Heat/moisture-treated starch, dried	15,73%
Skim milk powder	21,32%
Xanthan	1,12%
Common salt	4,51%
Citric acid granular	0,22%
Powdered onion and leek	5,56%
Sugar	2,50%
Saffron powder	0,08%
Various flavourings	16,03%

[0033] To prepare the creamy saffron soup 40g of this dry mixture was stirred into 200ml cold water, mixed and briefly

brought to the boil.

Example 3: Mushroom cream soup

[0034] A dry soup mix for a mushroom cream soup was made by mixing.

Creamer according to example 1-e	28,40%
Heat/moisture-treated starch, dried	14,76%
Skim milk powder	22,14%
Xanthan	1,05%
Common salt	4,22%
Citric acid granular	0,40%
Powdered onion and leek	5,18%
Sugar	1,10%
Powdered mushrooms and ceps	14,49%
Various flavourings	8,26%

[0035] To prepare the creamy mushroom soup 40g of this dry mixture was stirred into 200ml cold water, mixed and briefly brought to the boil.

Example 4: Tomato cream sauce

[0036] A dry sauce mixture for a creamy tomato sauce was made by mixing:

Creamer according to example 1-a	28,40%
Heat/moisture-treated starch, dried	14,76%
Tomato powder	36,63%
Xanthan	1,05%
Common salt	4,22%
Citric acid granular	0,40%
Powdered onion and leek	5,18%
Sugar	1,10%
Various flavourings	8,26%

[0037] To prepare the creamy tomato sauce 40g of this dry mixture was stirred into 200ml cold water, mixed and briefly brought to the boil.

Example 5: leek cream-style sauce

[0038] A dry sauce mixture for a leek cream-style sauce was made by mixing:

Creamer according to example 1-d	28,40%
Heat/moisture-treated starch, dried	14,76%
Leek powder	36,63%
Xanthan	1,05%
Common salt	4,22%
Citric acid granular	0,40%
Powdered onion and leek	5,18%
Sugar	1,10%
Various flavourings	8,26%

[0039] To prepare the creamy tomato sauce 40g of this dry mixture was stirred into 200ml cold water, mixed and briefly brought to the boil.

Example 8: creamer-cube

[0040] Following the process in patent EP 0779 039 a formed cream substitute was prepared by mixing 32,5 g spray dried cream powder (having a fat content of 75%) according to example 1-b with 32.5g spray dried creamer (including fat blend) on the basis of caseinate, having a fat content of 75% and the mixture was mixed in 35g of heated butter fat. The paste-like mass was extruded and formed into cubes of 7g each by a conventional molding machine.

[0041] The cubes so-obtained were equivalent in providing creaminess to that of an amount of at least 2 tablespoons of cream. This is the amount which is generally used for refining 250 ml of food. The cubes were dispersible in hot meals and were equivalent to the addition of cream as to whitening power, creaminess, sensation in the mouth and taste. The addition of the cubes unlike the addition of cream, did not lead to a dilution of the taste or the consistency of the meals. It was possible to store the cubes without cooling.

Claims

1. Particulates comprising 10-90% wt (preferably 15-80% wt) of triglycerides of fatty acids, which particulates are at least partly covered or encapsulated by 10-90% wt (preferably 20-85%) of an encapsulating or covering material, wherein the amount of H3 (triglyceride of 3 saturated fatty acids of 16 or more carbon atoms) and H2U (triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 cis-unsaturated fatty acid) taken together is at least 55% wt based on the total amount of triglycerides.

2. Particulates according to claim 1 wherein said amount of H3 + H2U is at least 65% wt based on the total amount of triglycerides.

3. Particulates according to claim 1-2, wherein the covering or encapsulation material comprises a protein and/or a carbohydrate.

4. Particulates according to claim 3, wherein the protein comprise a dairy protein, hydrolysed protein, gelatin, soy protein, or mixtures thereof.

5. Particulates according to claim 3, wherein the carbohydrate comprises a maltodextrin, a sugar, a sugar derivative, starch, chemically modified starch, physically modified starch, xanthan, guar, locust bean gum, alginate, carrageenan, polydextrose, or mixtures thereof.

6. Particulates according to claim 1-5, which particulates contain less than 30% wt (preferably less than 20% wt) of water.

7. Particulates according to claim 1-6, suitable as a creamer and/or whitener and/or non-dairy cream alternative in food compositions.

8. Particulates according to claim 1-7, wherein the amount of H3 (triglyceride of 3 saturated fatty acids of 16 or more carbon atoms) is at least 15% wt based on the total amount of triglycerides, preferably at least 20%.

9. Particulates according to claim 1-8, wherein the amount of H2U (triglyceride of 2 saturated fatty acids of 16 or more carbon atoms and 1 cis-unsaturated fatty acid) taken together is at least 40% wt based on the total amount of triglycerides.

10. Particulates according to claim 1-9, wherein the ratio H3 : H2U is between 1: 0.5 and 1:1.2.

11. Particulates according to claim 1-10, wherein the amount of H is between 60 and 75% wt based on total amount of fatty acids.

12. Particulates according to claim 1-11, wherein the amount of U is between 20 and 45% wt based on total amount of fatty acids

13. Particulates according to claim 1-12, wherein the amount of palmitic fatty acid (C16:0) based on the total amount of fatty acids is between 30 and 70% wt.

14. Particulates according to claim 1-13, wherein the particulates are in the shape of flakes, granules, powder, cube, pellet, or tablet.

15. Non-dairy cream alternative in the form of a cube, pellet or tablet comprising the particulates according to claim 1-14

16. Composition comprising 2-50% wt salt, 0-30% wt MSG, 0-20% wt herbs and/or spices, 0-30% wt vegetable particulates, 0-30% wt starch-based thickener and further comprising 0.1-30% wt of the particulates according to claim 1-15.

17. Composition according to claim 16, in the form of flakes, granules, powder or agglomerated or pressed to a cube, pellet, or tablet.

18. Composition according to claim 16-17, which is a soup- or sauce concentrate.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 02 07 9833

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
MUNICH	27 February 2003	Muller, I

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X : particularly relevant if taken alone
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EUROPEAN SEARCH REPORT

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A : technological background
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(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

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(81) Bestimmungsstaaten *(national)*: AE, AL, AM, AU, AZ, BA, BB, BG, BR, BY, CA, CN, CR, CU, CZ, DM, EE, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, RO, RU, SD, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Bestimmungsstaaten *(regional)*: ARIPO-Patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), eurasisches Patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI-Patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Veröffentlicht:
— *Mit internationalem Recherchenbericht.*

Zur Erklärung der Zweibuchstaben-Codes, und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

(54) Title: USE OF NANOSCALE STEROLS AND STEROL ESTERS

(54) Bezeichnung: VERWENDUNG VON NANOSKALIGEN STEROLEN UND STEROLESTERN

(57) Abstract: The invention relates to the use of nanoscale sterols and/or sterol esters with particle diameters of between 10 and 300 nm as food additives and as active agents for producing hypocholesterolaemic products. The invention is characterized by the particular fineness of the particles compared to sterols and sterol esters of the prior art. This results in quicker resorption through the serum with oral administration.

(57) Zusammenfassung: Vorgeschlagen wird die Verwendung von nanoskaligen Sterolen und/oder Sterolestern mit Teilchendurchmessern im Bereich von 10 bis 300 nm als Lebensmittelzusatzstoffe und Wirkstoffe zur Herstellung von hypocholesterinämischen Mitteln. Gegenüber Sterolen und Sterolestern des Stands der Technik bewirkt die besondere Feinheit der Partikel bei oraler Aufnahme eine raschere Resorption durch das Serum.

WO 01/00046 A1

Verwendung von nanoskaligen Sterolen und Sterolestern

Gebiet der Erfindung

Die Erfindung befindet sich auf dem Gebiet der Nanopartikel und betrifft die Verwendung von nanoskaligen Sterolen und Sterolestern als Lebensmittelzusatzstoffe.

Stand der Technik

Sterole und Sterolester stellen wichtige Rohstoffe sowohl für die Kosmetik und Pharmazie als auch für die Nahrungsmittelindustrie dar. So gibt es beispielsweise Hinweise dafür, daß Sterole, insbesondere pflanzliche Vertreter („Phytosterole"), in die Basalmembran der Haut eingebaut werden und über die Differenzierung der Hautzellen an die Hautoberfläche gelangen. Dies würde die pflegende und schützende Wirkung von Phytosterolen in der Hautkosmetik erklären. Die topische Anwendung von Sterolen führt auch zu einer erhöhten Hautfeuchtigkeit und einem gesteigerten Lipidgehalt. Hierdurch wird das Schuppungsverhalten der Haut verbessert und vorhandene Erytheme reduziert. Übersichten zu den Eigenschaften von Sterolen und Sterolestern in der Kosmetik finden sich beispielsweise von R.Wachter in **Parf.Kosm. 75, 755 (1994)** bzw. **Cosm.Toil. 110, 72 (1995)**. Eine weitere wichtige Eigenschaft von Phytosterolen und hier vor allem von Phytosterolestern ist ihre hypocholesterinämische Wirkung, d.h. ihre Fähigkeit, bei oraler Aufnahme, z.B. als Zusatzstoff zu Margarine, den Cholesteringehalt im Blut signifikant zu reduzieren, die schon 1953 von Peterson et al. in **J.Nutrit. 50, 191 (1953)** beschrieben wurde. In die gleiche Richtung weisen auch die Patentschriften **US 3,089,939, US 3,203,862** sowie die deutsche Offenlegungsschrift **DE-OS 2035069** (Procter & Gamble). Die Wirkstoffe werden üblicherweise Brat- oder Speiseölen zugesetzt und dann über die Nahrung aufgenommen, wobei die Einsatzmengen jedoch in der Regel gering sind und üblicherweise unter 0,5 Gew.-% liegen, um zu verhindern, daß die Speiseöle eintrüben oder die Sterole bei Zusatz von Wasser ausgefällt werden. Die Einarbeitung von Sitostanolestern zur Verminderung des Blutcholesteringehaltes in Margarine, Butter, Mayonnaise, Salatsaucen und dergleichen wird in der internationalen Patentanmeldung **WO 92/19640** (Raision) vorgeschlagen. In diesem Zusammenhang sei ferner auch auf die deutsche Patentanmeldung **DE-A1 19700796** (Henkel) verwiesen.

Die Wirkung der Sterole und Sterolester hängt stets mit der Geschwindigkeit zusammen, mit der die Verbindungen resorbiert werden. Hier besteht für die verfügbaren Stoffe des Stands der Tech-

nik noch ein erhebliches Verbesserungspotential. Die Aufgabe der vorliegenden Erfindung hat somit darin bestanden, die Aufnahme von Sterole und Sterolester bei oraler Aufnahme durch Bereitstellung neuer Anbietungsformen zu beschleunigen.

Beschreibung der Erfindung

Gegenstand der Erfindung ist die Verwendung von nanoskaligen Sterolen und/oder Sterolestern mit Teilchendurchmessern im Bereich von 10 bis 300 nm als Lebensmittelzusatzstoffe sowie als Wirkstoffe zur Herstellung hypocholesterinämischer Mittel.

Überraschenderweise wurde gefunden, daß sich die Resorption und hypocholesterinämische Wirkung von Sterolen und Sterolestern, insbesondere solchen auf Basis pflanzlicher Rohstoffe, signifikant steigern läßt, wenn diese in Form von Nanoteilchen, d.h. Partikeln mit einem mittleren Durchmesser im Bereich von 10 bis 300 und vorzugsweise 50 bis 150 nm vorliegen. Dabei ist zwischen zwei Ausführungsformen zu unterscheiden, nämlich der direkten Einarbeitung der Nanopartikel in die Lebensmittel und die Verkapselung der Partikel zur separaten oralen Aufnahme. Die Erfindung schließt ferner die Erkenntnis ein, daß die nanoskaligen Sterole bzw. Sterolester eine verbesserte Löslichkeit bzw. Dispergierbarkeit aufweisen, so daß es nunmehr möglich ist auch größere Einsatzmengen beispielsweise in Speiseölen klar und dauerhaft einzuarbeiten.

Sterole und Sterolester

Unter Sterolen (oder synonym Stenolen) sind tierische bzw. pflanzliche Steroide zu verstehen, die nur am C-3 eine Hydroxylgruppe, sonst aber keine funktionellen Gruppen tragen. In der Regel besitzen die Sterole 27 bis 30 Kohlenstoffatome und eine Doppelbindung in 5/6, gegebenenfalls 7/8, 8/9 oder anderen Positionen. Neben diesen ungesättigten Spezies kommen als Sterole auch die durch Härtung erhältlichen gesättigten Verbindungen in Frage, die als Stanole bezeichnet werden und von der vorliegenden Erfindung mitumschlossen werden. Ein Beispiel für ein geeignetes tierisches Sterol ist Cholesterol. Typische Beispiele für geeignete Phytosterole, welche aus anwendungstechnischen Gründen bevorzugt werden, sind beispielsweise Ergosterole, Campesterole, Stigmasterole, Brassicasterole sowie vorzugsweise Sitosterole bzw. Sitostanole und insbesondere β-Sitosterole bzw. β-Sitostanole. Neben den genannten Phytosterolen werden vorzugsweise deren Ester eingesetzt. Die Säurekomponente der Esters kann auf Carbonsäuren der Formel (I) zurückgehen.

R^1CO-OH
(I)

in der R^1CO für einen aliphatischen, linearen oder verzweigten Acylrest mit 2 bis 22 Kohlenstoffatomen und 0 und/oder 1, 2 oder 3 Doppelbindungen steht. Typische Beispiele sind Essigsäure, Propionsäure, Buttersäure, Valeriansäure, Capronsäure, Caprylsäure, 2-Ethylhexansäure, Caprinsäure, Laurinsäure, Isotridecansäure, Myristinsäure, Palmitinsäure, Palmoleinsäure, Stearinsäure, Isostearinsäure, Ölsäure, Elaidinsäure, Petroselinsäure, Linolsäure, konjugierte Linolsäure (CLA), Linolensäure, Elaeostearinsäure, Arachinsäure, Gadoleinsäure, Behensäure und Erucasäure sowie deren technische Mischungen, die z.B. bei der Druckspaltung von natürlichen Fetten und Ölen, bei der Reduktion von Aldehyden aus der Roelen'schen Oxosynthese oder als Monomerfraktion bei der Dimerisierung von ungesättigten Fettsäuren anfallen. Bevorzugt sind technische Fettsäuren mit 12 bis 18 Kohlenstoffatomen wie beispielsweise Kokos-, Palm-, Palmkern- oder Talgfettsäure. Besonders bevorzugt ist der Einsatz von Estern des β-Sitosterols bzw. β-Sitostanols mit Fettsäuren mit 12 bis 18 Kohlenstoffatomen. Diese Ester können sowohl durch direkte Veresterung der Phytostenole mit den Fettsäuren oder aber durch Umesterung mit Fettsäureniedrigalkylestern oder Triglyceriden in Gegenwart geeigneter Katalysatoren, wie z.B. Natriumethylat oder speziell auch Enzymen hergestellt werden [vgl. **EP-A2 0195311** (Yoshikawa)].

Herstellung von Nanopartikeln

Ein solches Verfahren zur Herstellung von Nanoteilchen durch rasche Entspannung von überkritischen Lösungen **(Rapid Expansion of Supercritical Solutions RESS)** ist beispielsweise aus dem Aufsatz von S.Chihlar, M.Türk und K.Schaber in **Proceedings World Congress on Particle Technology 3, Brighton, 1998** bekannt. Um zu verhindern, daß die Nanoteilchen wieder zusammenbacken, empfiehlt es sich, die Nanopartikel entweder unmittelbar nach der Herstellung den Lebensmitteln zuzusetzen oder aber die Ausgangsstoffe in Gegenwart geeigneter, d.h. insbesondere toxikologisch unbedenklicher Schutzkolloide oder Emulgatoren zu lösen und/oder die kritischen Lösungen in wäßrige und/oder alkoholische Lösungen der Schutzkolloide bzw. Emulgatoren zu entspannen, welche ihrerseits wieder gelöste Emulgatoren und/oder Schutzkolloide enthalten können. Geeignete Schutzkolloide sind dabei z.B. Gelatine, Chitosan, Casein, Gummi arabicum, Lysalbinsäure, Stärke sowie Polymere, wie etwa Polyvinylalkohole, Polyvinylpyrrolidone Polyalkylenglycole und Polyacrylate. Die bevorzugt zu verwendenden nanoskaligen Sterole und/oder Sterolester sind also die, die von einem toxikologisch unbedenklichen Schutzkolloid - und/oder einem Emulgator ummantelt vorliegen. Vorzugsweise kommen hier Gelatine, Chitosan oder deren Abmi-

schungen in Betracht. Üblicherweise werden die Schutzkolloide oder Emulgatoren in Mengen von 0,1 bis 20, vorzugsweise 5 bis 15 Gew.-% - bezogen auf die Sterole bzw. Sterolester - eingesetzt.

Ein weiteres geeignetes Verfahren zur Herstellung der nanoskaligen Teilchen bietet die **Evaporationstechnik**. Hierbei werden die Ausgangsstoffe zunächst in einem geeigneten organischen Lösungsmittel (z.B. Alkane, pflanzliche Öle, Ether, Ester, Ketone, Acetale und dergleichen) gelöst. Anschließend werden die Lösungen derart in Wasser oder einem anderen Nicht-Lösungsmittel, gegebenenfalls in Gegenwart einer darin gelösten oberflächenaktiven Verbindung gegeben, daß es durch die Homogenisierung der beiden nicht miteinander mischbaren Lösungsmittel zu einer Ausfällung der Nanoteilchen kommt, wobei das organische Lösungsmittel vorzugsweise verdampft. Anstelle einer wäßrigen Lösung können auch O/W-Emulsionen bzw. O/W-Mikroemulsionen eingesetzt werden. Als oberflächenaktive Verbindungen können die bereits eingangs erläuterten Emulgatoren und Schutzkolloide verwendet werden. Eine weitere Möglichkeit zur Herstellung von Nanoteilchen besteht in dem sogenannten **GAS-Verfahren** (Gas Anti Solvent Recrystallization). Das Verfahren nutzt ein hochkomprimiertes Gas oder überkritisches Fluid (z.B. Kohlendioxid) als Nicht-Lösungsmittel zur Kristallisation von gelösten Stoffen. Die verdichtete Gasphase wird in die Primärlösung der Ausgangsstoffe eingeleitet und dort absorbiert, wodurch sich das Flüssigkeitsvolumen vergrößert, die Löslichkeit abnimmt und feinteilige Partikel ausgeschieden werden. Ähnlich geeignet ist das **PCA-Verfahren** (Precipitation with a Compressed Fluid Anti-Solvent). Hier wird die Primärlösung der Ausgangsstoffe in ein überkritisches Fluid eingeleitet, wobei sich feinstverteilte Tröpfchen bilden, in denen Diffusionsvorgänge ablaufen, so daß eine Ausfällung feinster Partikel erfolgt. Beim **PGSS-Verfahren** (Particles from Gas Saturated Solutions) werden die Ausgangsstoffe durch Aufpressen von Gas (z.B. Kohlendioxid oder Propan) aufgeschmolzen. Druck und Temperatur erreichen nahe- oder überkritische Bedingungen. Die Gasphase löst sich im Feststoff und bewirkt eine Absenkung der Schmelztemperatur, der Viskosität und der Oberflächenspannung. Bei der Expansion durch eine Düse kommt es durch Abkühlungseffekte zur Bildung feinster Teilchen.

Gewerbliche Anwendbarkeit

Gegenüber Sterolen und Sterolestern des Stands der Technik bewirkt die besondere Feinteiligkeit der Partikel bei oraler Aufnahme eine raschere Resorption durch das Blutserum. Neben der in situ-Verkapselung der Nanopartikel ist es auch möglich, die Stoffe in üblichen Nahrungsmitteln zu lösen bzw. zu dispergieren, als da beispielsweise sind : Butter, Margarine, Diätnahrung, Frieröle, Speiseöle, Mayonnaisen, Salatdressings, Kakaoprodukte, Wurst und dergleichen. Die Einsatz-

menge der nanoskaligen Verbindungen liegt üblicherweise in der Größenordnung von 0,01 bis 5, vorzugsweise 0,1 bis 2 und insbesondere 0,5 bis 1 Gew.-% - bezogen auf die Mittel.

Beispiele

Herstellbeispiele. Zur Herstellung der nanoskaligen Sterole und Sterolester **(Beispiele 1 bis 5)** wurde zunächst Kohlendioxid einem Reservoir mit einem konstanten Druck von 60 bar entnommen und über eine Kolonne mit einer Aktivkohle- und einer Molekularsieb-Packung gereinigt. Nach der Verflüssigung wurde das CO_2 mit Hilfe einer Diaphragma-Pumpe bei einer konstanten Fördermenge von 3,5 l/h auf den gewünschten überkritischen Druck p verdichtet. Anschließend wurde das Lösungsmittel in einem Vorheizer auf die erforderliche Temperatur T1 gebracht und in eine Extraktionskolonne (Stahl, 400 ml) geleitet, welche mit dem Sterol bzw. Sterolester beladen war. Die resultierende überkritische, d.h. fluide Mischung wurde über eine lasergezogene Düse (Länge 830 µm, Durchmesser 45 µm) bei einer Temperatur T2 in eine Plexiglas Expansionskammer versprüht, die eine 4 Gew.-%ige wäßrige Dispersion eines Schutzkolloids enthielt. Das fluide Medium verdampfte und zurück blieben die im Schutzkolloid eingeschlossenen, dispergierten Nanopartikel. Zur Herstellung der Nanoteilchen gemäß **Beispiel 6** wurde eine 1 Gew.-%ige Lösung von Phytosterol in Aceton unter starkem Rühren bei 40°C und einem verminderten Druck von 40 mbar in eine 4 Gew.-% wäßrige Dispersion einer Mischung von Gelatine und Chitosan getropft. Das verdampfende Lösungsmittel wurde in einer Kühlfalle kondensiert, während die Dispersion mit den Nanopartikeln zurückblieb. Die Verfahrensbedingungen und der mittlere Partikelgrößenbereich (Bsp. 1 bis 5 photometrisch nach der 3-WEM-Methode, Bsp.6 über Laser-Rückstreuung bestimmt) sind in der nachfolgenden Tabelle 1 angegeben.

Tabelle 1
Nanopartikel

Bsp.	Sterol/Sterolester	Lsgm.	p bar	T1 °C	T2 °C	Schutzkolloid	PGB nm
1	Phytosterol*	CO_2	200	60	175	Gelatine	60-135
2	Phytosterol*	CO_2	180	70	160	Gelatine	75-125
3	β-Sitostanol	CO_2	200	85	180	Gelatine	75-130
4	β-Sitostenyllaurat	CO_2	200	85	175	Chitosan	55-140
5	β-Sitostanylstearat	CO_2	200	85	175	Gelatine/Chitosan (1:1)	60-150
6	Phytosterol*	-	-	-	-	Gelatine/Chitosan (1:1)	65-150

*) 58,1 Gew.-% β-Sitosterol, 29,8 Gew.-% Campesterol, 4,5 Gew.-% Stigmasterol; 3,8 Gew.-% Tocopherol; 0,4 Gew.-% Cholesterol; 0,3 Gew.-% Squalan; ad 100 Unverseifbares.

Anwendungsbeispiele. Es wurden Gelatinekapseln (Gewicht ca. 1,5 g) mit einem Gehalt von 5 Gew.-% β-Sitostanol bzw. β-Sitostanolester (Nanopartikel eingeschlossen in eine Gelatine- bzw. Chitosanmatrix bzw. nichtnanoskalige Handelsprodukte) sowie 0,5 Gew.-% radioaktiv markiertem Cholesterin hergestellt. Zur Untersuchung der hypocholesterinämischen Wirkung ließ man männliche Ratten (Einzelgewicht ca. 200 g) über Nacht fasten. Am folgenden Tag wurde den Versuchstieren jeweils eine zerkleinerte Gelatinekapsel mit etwas kochsalzhaltigem Wasser über eine Magensonde eingeführt. Nach 3, 6, 12, 24 und 48 h wurde den Tieren Blut abgenommen und der Gehalt an radioaktivem Cholesterin bestimmt. Die Ergebnisse, die den Mittelwert der Messungen von 10 Versuchstieren darstellen, sind in Tabelle 2 zusammengefaßt. Die Angaben zur Abnahme der Radioaktivität verstehen sich jeweils in Bezug auf eine Blindgruppe von Versuchstieren, denen lediglich Gelatinekapseln mit einem Gehalt von 20 Gew.-% Vitamin E und einer entsprechenden Menge radioaktiv markiertem Cholesterin verabreicht worden war. Die Beispiele 1 und 2 sind erfindungsgemäß, die Beispiele V1 und V2 dienen dem Vergleich.

Tabelle 2
Hypocholesterinämische Wirkung

Bsp.	Phytosterol(ester)	Radioaktivität [%-rel.]				
		nach 3 h	nach 6 h	nach 12 h	nach 24 h	nach 48 h
V1	β-Sitostanol*	93	83	75	50	32
V2	β-Sitostanylstearat*	90	80	71	44	26
1	Nano-β-Sitostanol**	88	77	69	44	27
2	Nano-β-Sitostanylstearat***	85	74	66	37	21

*) Handelsprodukte **) gem. Herstellbeispiel 3 ***) gem. Herstellbeispiel 4

Patentansprüche

1. Verwendung von nanoskaligen Sterolen und/oder Sterolestern mit Teilchendurchmessern im Bereich von 10 bis 300 nm als Lebensmittelzusatzstoffe.

2. Verwendung nach Anspruch 1, **dadurch gekennzeichnet**, daß man Phytosterole oder deren Ester einsetzt.

3. Verwendung nach den Ansprüchen 1 und 2, **dadurch gekennzeichnet**, daß man Sitosterole oder deren Ester einsetzt.

4. Verwendung nach mindestens einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß man nanoskalige Sterole und/oder Sterolester einsetzt, die man erhält, indem man

   (a) die Ausgangsstoffe unter überkritischen oder nahekritischen Bedingungen in einem geeigneten Lösungsmittel löst,
   (b) die fluide Mischung über eine Düse in ein Vakuum, ein Gas oder eine Flüssigkeit entspannt, und
   (c) das Lösemittel dabei gleichzeitig verdampft.

5. Verwendung nach mindestens einem der Ansprüche 1 bis 4, **dadurch gekennzeichnet**, daß man Nanopartikel einsetzt, welche von einem toxikologisch unbedenklichen Schutzkolloid ummantelt vorliegen.

6. Verwendung nach Anspruch 5, **dadurch gekennzeichnet**, daß man als Schutzkolloid Gelatine und/oder Chitosan einsetzt.

7. Verwendung nach mindestens einem der Ansprüche 1 bis 6, **dadurch gekennzeichnet**, daß man die Sterole und/oder Sterolester in Mengen von 0,01 bis 5 Gew.-% - bezogen auf die Mittel - einsetzt.

8. Verwendung nach mindestens einem der Ansprüche 1 bis 7, **dadurch gekennzeichnet**, daß man die Sterole und/oder Sterolester in Butter, Margarine, Diätnahrung, Frittierölen, Speiseölen, Mayonnaisen, Salatdressings, Kakaoprodukten oder Wurst einsetzt.

9. Verwendung von nanoskaligen Sterolen und/oder Sterolestern mit Teilchendurchmessern im Bereich von 10 bis 300 nm als Wirkstoff zur Herstellung von hypocholesterinämischen Mitteln.

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/EP 00/05537

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A23L1/30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A23L A23D A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ, FSTA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 897 671 A (UNILEVER PLC ;UNILEVER NV (NL)) 24 February 1999 (1999-02-24) claims 1,4-6,10,13,20,33; examples 1-6 page 3, line 39-42,51-55 page 4, line 2-4,12,13,27,28 page 5, line 20-22,26,27,38-41 page 6, line 9-16,23,24	1-3,5-9
Y	page 7, line 35,36 ---	1-9
X	EP 0 087 993 A (DIOR CHRISTIAN PARFUMS) 7 September 1983 (1983-09-07) page 3, line 29 -page 4, line 10 page 11, line 35 -page 12, line 4 page 16, line 6-17 page 19, line 1-5,15-19	1-3,5,7
A	claims 10,11; examples 1,10,16 --- -/--	2,4,6,8, 9

[X] Further documents are listed in the continuation of box C. [X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
4 October 2000	11/10/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Tallgren, A

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 00/05537

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	page 25, paragraph 1 -page 26, paragraph 3	4
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A	"Micronization of organic solids by rapid expansion of supercritical solutions" STN CHEMICAL ABSTRAC, XP002126942 abstract	1,4
A	"Effect of process parameters on particles obtained by rapid expansion of supercritical solutions" STN CHEMICAL ABSTRAC, XP002126943 abstract	1,4

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INTERNATIONALER RECHERCHENBERICHT

Internales Aktenzeichen
PCT/EP 00/05537

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
IPK 7 A23L1/30

Nach der internationalen Patentklassifikation (IPK) oder nach der nationalen Klassifikation und der IPK

B. RECHERCHIERTE GEBIETE

Recherchierter Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)
IPK 7 A23L A23D A61K

Recherchierte aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen:

Während der internationalen Recherche konsultierte elektronische Datenbank (Name der Datenbank und evtl. verwendete Suchbegriffe)
EPO-Internal, WPI Data, PAJ, FSTA

C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
X	EP 0 897 671 A (UNILEVER PLC ;UNILEVER NV (NL)) 24. Februar 1999 (1999-02-24) Ansprüche 1,4-6,10,13,20,33; Beispiele 1-6 Seite 3, Zeile 39-42,51-55 Seite 4, Zeile 2-4,12,13,27,28 Seite 5, Zeile 20-22,26,27,38-41 Seite 6, Zeile 9-16,23,24	1-3,5-9
Y	Seite 7, Zeile 35,36	1-9
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A	Ansprüche 10,11; Beispiele 1,10,16	2,4,6,8, 9

-/--

[X] Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen

[X] Siehe Anhang Patentfamilie

* Besondere Kategorien von angegebenen Veröffentlichungen :
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"&" Veröffentlichung, die Mitglied derselben Patentfamilie ist

Datum des Abschlusses der internationalen Recherche	Absendedatum des internationalen Recherchenberichts
4. Oktober 2000	11/10/2000

Name und Postanschrift der Internationalen Recherchenbehörde	Bevollmächtigter Bediensteter
Europäisches Patentamt, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Tallgren, A

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INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
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C (Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
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P,Y	WO 00 21490 A (HOLLENBROCK MARTINA ;FABRY BERND (DE); HENKEL KGAA (DE); FOERSTER) 20. April 2000 (2000-04-20) Seite 2, Absatz 2 -Seite 4, Absatz 1 Ansprüche 1-7; Beispiel 1; Tabelle 1	1-9
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A	Ansprüche 1,7,9; Beispiele 1,4; Tabelle 1 Seite 6, Zeile 6-15,25-30 Seite 7, Zeile 14-23 Seite 8, Zeile 5-10	6,8,9
A	"Micronization of organic solids by rapid expansion of supercritical solutions" STN CHEMICAL ABSTRAC, XP002126942 Zusammenfassung	1,4
A	"Effect of process parameters on particles obtained by rapid expansion of supercritical solutions" STN CHEMICAL ABSTRAC, XP002126943 Zusammenfassung	1,4

1

INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
PCT/EP 00/05537

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(54) Aqueous dispersions or suspensions

(57) Aqueous dispersions of plant sterols and other high melting lipids. The dispersions are useful in spreads and other food products. The dispersions provide structure to the food products and their use can permit minimization or elimination of saturated fats and trans fatty acids. The invention is also directed to a process for making the dispersions and to water and fat continuous spreads and other food products including the high melting lipids.

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Description

Background of the Invention

[0001] Hydrogenation, fractionation and interesterification are the most common processes utilized within the fats and oils industry to modify the chemical and/or physical properties of conventional triglyceride fats or other lipids to improve their utility and functionality, e.g. to modify melting points, increase hardness or the solid fat content, increase oxidative stability, etc. These processes may be used individually or can be combined to produce fats and oils with very specific characteristics. When employing these processes to increase hardness or the solid fat content in the simplest of terms, these processes rely on the reduction of unsaturation or the increase of saturation and/or the formation of trans fatty acids.

[0002] Considerable attention has been drawn in recent years to the relatively high total fat, saturated fat and trans fat content of the typical diet.

[0003] Related health issues which have been greatly publicized are elevated cholesterol levels and low HDL/LDL cholesterol ratios reportedly resulting from ingestion of saturated fats and cholesterol. And, in addition to saturated fats, some reports have implicated trans fatty acids, which are generated when liquid oils are partially hydrogenated to increase their solid fat content.

[0004] Phytosterols are sterols found in plants. While these compounds have long been touted for their cholesterol lowering effects, they tend to be very high melting compounds (melting points around 150°C), and they are difficult to formulate into consumer food products due to poor solubility in fats and immiscibility in water. This solubility problem has been partially mitigated by esterification of the sterol. Nevertheless, this still limits use of sterols to food compositions with moderate to high fat contents.

[0005] U.S. Patent No. 5,156,866 discloses sterols used in chewing gums. An emulsifier is added to decrease viscosity.

[0006] U.S. Patent No. 5,445,811 is directed to phytosterols used in an oil-in-water emulsion intended for intravenous administration as a contrasting agent for visibilizing the presence of tumors. Synthetic emulsifiers of the type normally used for preparing oil-in-water emulsions are said to be of particular interest. The weight ratio between emulsifier and cholesterol or phytosterol can range between 1:1 and 1:2. In Example 2, 0.06g cholesterol is used per 100 ml of mixture. The mixture is homogenized and autoclaved at 121°C. In Example 1, 2g of cholesterol are used and the mean particle size of the emulsion is 0.25 mu m. In Example 6, 1.5g of cholesterol per 100ml mixture are used. It is said that the mean size of the emulsion particles will preferably be beneath 1 mu m.

[0007] U.S. Patent No. 5,244,887 is directed to the use of plant stanols to reduce cholesterol absorption from foods. It is said that the greatest effectiveness is obtained when the stanols are evenly distributed in finely divided form throughout the food product or beverage. This can be accomplished by dissolving the stanols in a solubilizing agent such as vegetable oil, monoglyceride, diglycerides, tocopherols, and mixtures thereof and making suspensions or emulsions of the solubilized stanols in carriers such as water, alcohol, polyols and other edible compounds or by suspension of the stanols in an emulsion. Solubilizing agents such as monoglycerides and diglycerides are mentioned. A preferred food additive is said to comprise, in addition to 25% stanols, 74.8% vegetable oil and tocopherol. The compounds of the invention are used as food additives to foods such as meats, eggs, and dairy products. The stanols are said to remain in solution or uniformly suspended.

[0008] U.S. Patent No. 4,150,850 mentions low fat margarine products having less than 60 wt. % fat. The invention is directed to a shelf stable mix suitable for consumer preparation of a spreadable butter substitute having from about 20-80% oil and from about 10 to about 60% water. A water-in-oil emulsifier is included. Preferred emulsifiers are phytosterols. In preparing the solid form of the mix, the emulsifier and hard fat having a melting point of from about 29°C to about 66°C are formed together as a mixture and solidified. The emulsifier is then added to the melted fat and mixed until a clear solution is obtained.

[0009] U.S. Patent No. 3,965,939 is directed to edible cooking and salad oil compositions having enhanced hypocholesterolemic properties including plant sterols. The limited solubility of plant sterols in any solvent system is noted. A solubilizing agent to solubilize the plant sterol in the oil is selected from the group consisting of fatty acids, monoesters of fatty acids with polyhydric alcohols and alkanols. The invention is said also to contemplate peanut butter, mayonnaise, ice cream and margarine spreads. A solubilizing agent may be selected from a group including simple esters of fatty acids such as monoglycerides.

[0010] U.S. Patent No. 5,502,045 is directed to a beta-sitostanol fatty acid ester or mixture thereof which lowers cholesterol levels. In Example 5 a beta-sitostanol ester mixture is added to the fatty part of a conventional soft margarine.

[0011] Ong, U.S. Patent No. 4,195,084 is directed to a pharmaceutical preparation comprising a taste-stable aqueous suspension of tall oil sitosterols.

[0012] Beta sitosterol is described as being the most effective of the sterols for lowering serum cholesterol. Because of certain physical properties of the sterols, it is said not to have been practical to provide a pharmaceutical suspension

for oral administration which contains much more than 20 w/v of sitosterols. It is said that in order for sitosterols to be the most effective in lowering serum cholesterol the medicament must reach the gastrointestinal tract in a finally divided dispersed state

[0013] Ong also reports that sitosterols do not lend themselves readily to incorporation into an aqueous preparation for oral administration that has a pleasant mouthfeel.

[0014] The Ong invention is directed to an aqueous pharmaceutical suspension comprised of finally divided tall oil sitosterols, a pharmaceutically acceptable chelating agent, sodium carboxymethylcellulose, sorbitol, a pharmaceutically acceptable surfactant, simethicone and water. The product is said to have an acceptable taste and mouthfeel that does not change over an extended storage period.

[0015] The pharmaceutical suspension of Ong may contain up to about 25 w/v of finally divided tall oil sitosterols. At about 20% tall oil sitosterols, the suspension is said not to be excessively viscous and is relatively easy to pour, having both good physical and chemical stability. A tall oil sitosterols preparation is said to develop no taste change after one year of shelf storage as compared with bitter taste development within two weeks at room temperature for a suspension of tall oil sitosterols not within the Ong invention. It is said that tall oil sitosterols are very hydrophobic and stubbornly resist wet wetting. Vigorous continuous agitation is said to be required to disperse the tall oil sitosterols in the vehicle.

[0016] The Ong phytosterol are said to be ground to a mean particle size of 25 microns or below by use of an air mill, high energy hammermill or air filtration mill under refrigeration or through the use of finely ground dry ice.

[0017] EP 289 636 discloses an emulsified or solubilized sterol composition wherein the sterols are emulsified or solubilized in an aqueous solution of polyhydroxy compounds containing sucrose fatty acid esters and/or polyglycerol fatty acid esters or liquid polyhydroxy compounds. A considerably high shelf stability is said to result and the invention is said to be extremely useful in various products including food, cosmetics, drugs and agricultural chemicals.

[0018] Sterols are described as high melting compounds which are hardly soluble in water and have a melting point of approximately 150°C. It is said to be difficult to obtain a stable, emulsified or solubilized composition. Beta-sitosterol is mentioned among the sterols which may be used in the '636 invention.

[0019] The compositions are prepared by, for example, adding sucrose ester and/or polyglycerol fatty acid ester to an aqueous solution of polyhydroxy compound, heating the mixture to 50 to 60°C, adding powdery sterols, stirring the obtained mixture at 50-90°C to dissolve the sterols and diluting the obtained solution if required. The compositions are said to show stable emulsification or solubilization without causing any separation of the sterols.

[0020] "Effect of Plant Sterols on Lipids and Atherosclerosis", Pollack, O.J., Pharmac. Ther., 31, 177-208 (1985) is reported in U.S. Patent No. 5,244,887 as suggesting the inclusion of plant sterols such as beta-sitosterol in such foods as butter and margarine to counteract not only the cholesterol in butter, but all other dietary cholesterol and cholesterol from non-dietary sources available for absorption and reabsorption.

Summary of the Invention

[0021] The present invention is directed to the discovery that phytosterols and other high melting lipids can be used to impart structure to water and fat continuous spreads and other products including aqueous phases. As mentioned above, U.S. Patent No. 3,865,939 discloses the well known difficulties in solubilizing plant sterols in any solvent system.

[0022] In the present invention, an aqueous dispersion of phytosterol or other high melting lipid is formed which is finely dispersed and stable, wherein the phytosterols or other high melting lipid serve to structure the aqueous dispersion. This is particularly useful in water and fat continuous spreads and other food systems or compositions, wherein the aqueous phase is structured by the phytosterol or other high melting lipid.

[0023] By using phytosterols or other high melting lipids as structuring agents, it is possible to avoid or minimize the use of saturated fat and other traditional structure-imparting ingredients in food products. For example, the presence of conventional thickeners such as gelatin and xanthan gum can be minimized or avoided. Moreover, even the inclusion of partially hydrogenated fat, which generally include trans fatty acids, can be avoided by use of phytosterols or other high melting lipids as structuring agents. At the same time, an added benefit where phytosterols are used is the reported cholesterol lowering effect of phytosterols. An even further benefit is the reduced calories which result from using high melting lipids as structuring agent since phytosterols and some other high melting lipids are barely or completely non-digestible.

[0024] Although a particularly advantageous use of the aqueous dispersions of the invention is in the preparation of water continuous low or no fat spreads, other food products can benefit from inclusion of the aqueous dispersions or suspensions according to the invention. These include fat continuous spreads which may be either vegetable oil or butter fat based, bi-continuous spreads, dressings, beverages, dairy products, such as milk, cheeses, yogurt, non-dairy coffee whiteners, beverages, ice cream, and confections such as candy or chocolate.

[0025] The aqueous dispersions are useful as such in the preparation of foods and other products. Indeed, with the dispersions and process of the invention, it is possible to make very concentrated phytosterol dispersions or suspensions, which have a number of important functional uses such as structuring, bodying and bulking agents, and whiten-

ing/opacity providers, especially in reduced and low fat foods.

[0026] The phytosterols and other high melting lipids are preferably present in the dispersion or suspension as very finely divided particles having a size of 15 microns or less, preferably 10 microns or less. The dispersion or suspension also include a non-phytosterol emulsifier. The inclusion of a non-sterol emulsifier may be omitted where sterols have been esterified with highly hydrophilic compounds such as citric acid, tartaric acid, for example. Such a chemical modification to phytosterols or other sterols would preclude the need to use a separate emulsifier of the type such as mono and diglyceride and polysorbate 60 in preparing the dispersion or suspension. The weight to weight ratio of emulsifier to phytosterol or other high melting lipid in the aqueous phase is less than 1:2, preferably less than 1:2.25, most preferably less than 1:3. Moreover, the aqueous phase dispersion or suspension according to the invention need not to include a large amount of triglyceride fat or other non-high melting lipid. However these non-high melting lipids can be present as well. If present the weight to weight ratio of non-phytosterol, non-high melting lipid to phytosterol or other high melting lipid in the aqueous phase is preferably less than 1:6, more preferably less than 1:8. Preferred phytosterols are Beta sitosterol, campesterol, stigmasterol, brassicasterol and ergosterol.

[0027] A particularly advantageous use of the aqueous dispersions of the invention is in an oil-in-water-in-oil spread. In such spreads, the phytosterols or other high melting lipids are preferably used to structure both the continuous oil phase (external phase) and the dispersed aqueous phase. It has been found that such spreads including a continuous oil phase having phytosterols or other high melting lipids, a dispersed aqueous phase having phytosterols or other high melting lipids and a second oil phase dispersed in the aqueous phase have a reduced tendency toward oil separation and therefore an increased product stability. Preferably the continuous fat phase comprises from 0.5 to 5 wt. % phytosterol or other high melting lipid and the dispersed aqueous phase comprises from 2 to 15 wt. % phytosterol or other high melting lipid.

[0028] The aqueous dispersions or suspensions of the invention are preferably prepared by melting the phytosterol (or other high melting lipid) and the emulsifier and dispersing the molten phytosterol (or other high melting lipid) and emulsifier in water under shear. While not wanting to be limited by theory, it is believed that the step of melting the high melting phytosterols with surfactant prior to dispersing in water with or without surfactant contributes importantly to the ability to prepare a very fine dispersion with the use of high shear mixing or homogenization of the phytosterol or other high melting lipid. Preferably the phytosterols or other high melting lipid in the present process and dispersions have been reduced to a size of 15 microns or lower, preferably 10 microns or lower.

Detailed Description of the Invention

[0029] The high melting lipid of the invention is preferably a phytosterol, i.e. plant sterols, such as alpha sitosterol, beta sitosterol, stigmasterol, ergosterol and campesterol, alpha spinosterol and brassicasterol. Although the foregoing are some of the more important phytosterols, at least 44 phytosterols have been identified and it will be apparent to one of ordinary skill that many of these will be appropriate for the present invention. Phytosterols are identified in Bean (1993) phytosterols in "*Advances in Lipid Research*", pages 193-218, Paoletti, and Kiritchevsky, (Eds) Academic Press, NY, the disclosure of which is incorporated herein by reference. The disclosure of "Effect of Plant Sterols on Lipids and Atherosclerosis", Pollack, O.J., Pharmac. Ther., 31, 177-206 (1985) mentioned above is also incorporated by reference herein.

[0030] Many sources of phytosterols are known. Among sources are disclosed in Pollak "*Effect of Plant Sterols on Serum Lipids and Atherosclerosis*", Pharm. Ther. Vol. 31, pp. 177-206, 1985, the disclosure of which is hereby incorporated by reference. See especially Table 7 on page 202. Among the more important sources are rice bran, corn bran, corn germ, wheat germ oil, corn oil, safflower oil, oat oil, olive oil, cotton seed oil, soybean oil, peanut oil, black tea, orange juice, valencia, green tea, Colocsia, kale, broccoli, sesame seeds, shea oils, grapeseed oil, rapeseed oil, linseed oil, canola oil, tall oil from wood pulp and other resinous oil from wood pulp.

[0031] While particular benefit is obtained when the invention is used to emulsify or solubilize phytosterols or their esters, especially those which have been shown to have a cholesterol lowering benefit, zoosterols, fungal, algeal and microbial sterols, and other high melting sterols and other lipids may also be used, as appropriate. Among the known zoosterols are cholesterol, 24-methylene-cholesterol, 7,22-dehydroxycholesterol and desmosterol.

[0032] It will generally be desirable to employ high purity and practical grade sterols and other high melting lipids which are suitable for ingestion by humans.

[0033] In addition to zoosterols, phytosterols and other sterols, it is believed that the present invention may be used advantageously with other high melting, water insoluble lipids. The high melting, water insoluble, sterols and other lipids of the invention have a melting point within the range of 75-200°C. Especially preferred are lipids with melting points of 100-200°C, and especially from 125-175°C.

[0034] Other classes of high melting lipids, in addition to the sterols, which may be used herein are the waxes in particular, carnauba wax, bees wax, waxes and wax esters from vegetable oil sources, but also sterols, sterolesters, stanols, stanolesters, hardened vegetable oils, saturated triglyceride fractions of vegetable oils, mono- and diglycerides

can be used. The phytosterols however are the preferred materials.

[0035] Melting point may be measured by known methods such as the AOCS capillary tube method and/or the Thomas-Hoover Uni-Melt melting point apparatus, ex. Thomas Scientific, Swedesboro, NJ

[0036] The invention is used to greatest advantage when employing phytosterols and other sterols which have not been esterified. Phytosterols which have been esterified are more readily dissolved in oil phases and do not face to as great an extent the problem of solubilization and dispersion in food products having continuous fat phases. However, it may be appropriate under certain circumstances to utilize esterified phytosterols and other high melting lipids, so long as they are high melting, immiscible in water and fall in the desired melting temperature range of 75-200°C.

[0037] While the invention has been described as being particularly relevant to sterols, the invention may also be applied to their hydrogenated counterparts, such as phytostanols and to other chemically modified sterols. Chemical modifications include in addition to complete and partial hydrogenation, esterification, including interesterification. Examples of phytostanols include campestanol, 22,23 dihydrobrassicastanol, beta-sitostanol and clionastanol. Fatty acids esterified to the sterols include long and short chain fatty acids, i e. C_1-C_{22}.

[0038] The phytosterols or other high melting lipids will suitably comprise about 1 to about 75% of the aqueous dispersions, preferably from 5 to 40 wt. %. However for other applications other amounts can also be used.

[0039] Many emulsifiers may be used to disperse the phytosterols or other high melting lipids. Preferred emulsifiers include polyglycerol esters and tweens, especially polysorbate 60. Other examples of emulsifiers which may be used include mono- and diglycerides, e.g., Myverol 18-04 available from Quest International, Hoffman Estates, Ill., sodium stearoyl lactylate, and polysorbates. Most preferred are oil-in-water emulsifiers.

[0040] The aqueous dispersions according to the invention may include, in addition to the phytosterols or other high melting lipids, ingredients such as the following: water, salt, flavors, preservatives, gums, starches, gelatin, milk and milk protein, colors, acidulants such as citric acid. Obviously, the aqueous dispersion may contain ingredients destined for the ultimate food product to be prepared from the dispersion.

[0041] The aqueous phytosterol and other high melting lipid dispersion of the invention may be concentrated by centrifugation, decantation, evaporation or other methods.

[0042] The concentration of the phytosterols or other high melting lipids in the aqueous dispersion can range from 0.1 to 99 wt. %, especially from 5 to 75, more particularly from 10 to 50.

[0043] As indicated above, a preferred method of preparing the aqueous dispersion involves mixing molten phytosterol or other high melting lipid together with molten surfactants in water. Preferably the surfactant level in the molten phytosterol phase is 1-20 wt. %. Alternatively, the molten surfactant can be incorporated separately into the aqueous phase.

[0044] A bicontinuous spread can suitably be made by dispersing a liquid oil into an aqueous dispersion of high melting lipid to form a first dispersion and then dispersing said first dispersion into a high melting lipid-containing oil phase. In that instance a product is made, wherein the continuous oil phase comprises 1-25 wt.% high melting lipids, the aqueous phase comprises 1-25 wt.% of one or more high melting lipid and the continuous oil phase comprises 25-95 wt. % of the spread and the internal oil phase comprises from 0 to 70 wt. % of the spread, the aqueous phase comprising 0 to 75 wt. % of the spread.

[0045] Among the foods in which the dispersions of the invention can provide structuring include water continuous spreads, fat continuous spreads, bicontinuous spreads, dressings, drinks, dairy products (such as milk, yogurt, cheese, cream cheese) dry mixes, powdered non-dairy coffee whiteners, milkshake mixes, confections, ice creams, instant milks, cake mixes and other food and pharmaceutical preparations.

[0046] As indicated above, the aqueous dispersions can be used to structure water continuous spreads or oil-in-water-in-oil spreads. In such case, the phytosterols preferably are present to structure both the continuous oil external phase and the dispersed aqueous phase.

[0047] Although not required, if desired the aqueous dispersions of the invention can be used in conjunction with other structuring agents in the spreads and other food products of the present invention. Such structuring agents include the mesomorphic phases of edible surfactant disclosed in WO 92/09209, the disclosure which is incorporated herein by reference.

[0048] Spreads according to the embodiment generally contain from less than 85% by weight of edible triglyceride materials. Suitable edible triglyceride materials are for example disclosed in *Bailey's Industrial Oil and Fat Products* (1979). In higher fat spreads, the level of triglyceride material will generally be more than 60% and less than 80%, preferably from 70 to 79% by weight. In spreads of reduced fat content the level of triglycerides will generally be from 30-60%, more generally from 35 to 45% by weight. In very low fat spreads the level of triglycerides will generally be from 0-40%, for example 30%, 25%, 20% or even 10% or about 0%. Other fatty materials, for example sucrose polyesters may be used as a replacement for part or all of the triglyceride material. Preferred water continuous spreads comprise 0-85 wt. % fat and 100-15 wt.% continuous aqueous phase. Preferred fat continuous spreads comprise 15-80 wt. % fat and 85-20 wt. % water phase.

[0049] The phytosterol or other high melting lipid material for use in spreads is preferably used at a level of from 5-50

wt. % of the aqueous phase, more preferred from 10-50%, most preferred from 20 to 40 % by weight. Spreads may comprise additional surfactants to those used to disperse the high melting lipid, for instance, monoglycerides and lecithins, ionic edible surfactant such as lactylated fatty acid salts and phophatidic acid.

[0050] The water phase of the water continuous spread can suitably contain a non-phytosterol emulsifier and high melting lipid in a w/w-ratio of less than 1.2. The aqueous phase can also contain a non-high melting, non-phytosterol lipid. In that instance the w/w ratio of the non-phytosterol lipid to the high melting phytosterol is less than 1:6. The fat phase of fat continuous spreads can include structuring lipids, selected from high melting lipids and not high melting lipids. The high melting lipids preferably being phytosterols.

[0051] In addition to the above mentioned ingredients, spreads in accordance with the invention may optionally contain further ingredients suitable for use in spreads. Examples of these materials are gelling agents, thickening agents, sugars, eg sucrose or lactose, or other sweetener materials, EDTA, spices, salt, bulking agents, flavoring materials, coloring materials, proteins, acids etc. Suitable biopolymer materials which may be included in spreads include, for example, milk protein, gelatin, soy protein, xanthan gum, locust bean gum, hydrolyzed starches (for example Paselli SA2 and N-oil), and microcrystalline cellulose. Other gelling and thickening agents which may be used include but are not limited to carrageenan, pectin, gellan gum, agar, guar, alginate, maltodextrin, native and modified starches, and pregelatinized starches. Appropriate aqueous and fat phase ingredients are found in Cain et al. US Patent No. 4,917,915 and Norton et al. US Patent Nos. 5,194,285 and 5,151,290, the disclosures of which are hereby incorporated by reference.

[0052] Various sources for the gelling agents include plants, including marine plants, microorganisms, and animals. The amount of biopolymer, if any, in spreads of the invention is dependent on the desired degree of gelling and the presence of other ingredients in the composition. The amount of gelling agent may lie between 0 and 30%, mostly between 0.1 and 25% based on the weight of the aqueous phase of the spread. If hydrolyzed starches are present their level may be from 2-20%; other gelling agents may be used at levels of up to 10%, mostly 1-7%, most preferred 2-5% all percentages being based on the weight of the aqueous phase. Particularly preferred are combination of say 2-15% hydrolyzed starch and 0.5-5% of other gelling materials, especially gelling materials including gelatin.

[0053] In addition to or in combination with the above, the aqueous phase of spreads or other foods may include the following ingredients: dairy ingredients such as buttermilk, skim milk, milk, salt, acidulants, such as lactic acid and citric acid, butter, yogurt, whey, caseinate, milk proteins, vegetable proteins, vitamins and preservatives such as potassium sorbate and sodium benzoate.

[0054] The balance of the spread composition is generally water, which may be incorporated at levels of up to 99.9% by weight, more general from 10 to 98%, preferably from 20 to 97% by weight. Spreads according to the invention may be fat and/or water continuous.

[0055] Where the spread or other food product of the invention includes a fat phase, the composition of the fatty phase preferably comprises one or more vegetable oils, preferably sunflower oil, soybean oil, rapeseed oil, canola oil, corn oil, peanut/groundnut oil and the like. Although not generally preferred, if desired, dairy and other animal fat may also be used. Dairy, other animal fat sources and miscellaneous fat sources include milk (milk fat), buttermilk, fish oil, lard and tallow. If desired, the fat may be hydrogenated, fractionated and/or interesterified, but again it will usually be less desirable to include hydrogenated fat, which will be saturated and which may include trans fatty acids.

[0056] While the fat that is applied in these fat based food products can be any fat, such as dairy fat and/or vegetable fat, if fat is present, for health reasons the use of one or more vegetable fat sources is preferred. In particular, the use of liquid fats is preferred. The fat can be one single fat or a blend. The use of fat compositions comprising a considerable amount of PUFA (poly unsaturated fatty acid) rich triglycerides in addition to the use of the sterol/sterol ester mixture is in particular considered highly beneficial. For example, oils of sunflower, safflower, rapeseed, linseed, linola and/or soybean can be used in a preferred embodiment. Also the fat compositions mentioned in Netherlands patent documents no

NL 143115, NL 178559, NL 155436, NL 149687, NL 155177, European patent documents EP 41303, EP 209176, EP 249282, and EP 470658, the disclosures of which are incorporated by reference herein, are highly suitable.

[0057] If a fat blend is used, it is preferred that it comprises at least 30%, and more preferred at least 45% of polyunsaturated fatty acid moieties, based on the total weight amount of the fat in the fat based food product. So a strong effect on the cholesterol lowering effect is obtained if use is made of an optimal ratio of sterol and sterol-esters as set forth in this application in a food product in which a fat blend comprising at least 30 wt. % of PUFA rich triglycerides is used.

[0058] Where butterfat is used for preparing spreads of the invention, or where the spreads are butter, it is preferred that the amount of phytosterol is in the range of 5-15%, preferably 10-15%. As the consumption of butter is considered less beneficial for consumers health, the present invention is in particular suitable for making butter or butter-melanges containing spreads, as the negative effect associated with the butter consumption can be minimized or even reversed.

[0059] Generally, dressings or mayonnaise are oil in water emulsions. The oil phase of the emulsion generally is 0 to 85% by weight of the product. For higher fat products the level of triglycerides is generally from 60-85%, especially from 65-80% by weight. For salad dressings the level of fat is generally from 10-60%, more preferred from 15 to 40%. Low

or no-fat containing dressings may for example contain triglyceride levels of 0, 5, 10 or 15% by weight.

[0060] Other fatty materials such as for example polyol fatty acids ester may be used as a replacement for part or all of the triglyceride materials in the dressings or other foods of the invention.

[0061] The level of edible surfactant material in the dressing will generally be from 0.1 to 15%, more preferred from 1-10%, most preferred from 2 to 8% by weight. Preferably the level of nonionic edible surfactant is from 0.1 to 15%, more preferred, 0.5-10%, most preferred 1 to 8% by weight. Especially preferred are monoglycerides as nonionic edible surfactants. Preferably the level of ionic edible surfactant is form 0 to 5%, more preferred 0.05 to 2%, most preferred 0.1 to 0.5% by weight.

[0062] Dressings are in general low pH products with a preferred pH of from 2-6, more preferred 3-5, for example about 3.5. For the use in dressings the preferred anionic is the diacetyl tartaric ester of monoglycerides (in the examples Admul DATEM 1935 ex. Quest Int. has been used). Also an anionic phospholipid such as phosphatidic acid can be applied.

[0063] In addition to the above mentioned ingredients dressings in accordance with the present invention optionally may contain one or more other ingredients which may suitably be incorporated into dressings and/or mayonnaise. Examples of these materials are emulsifiers, for example
egg-yolk or derivatives thereof, stabilizers, acidifiers, biopolymers, for example hydrolysed starches and/or gums or gelatin, bulking agents, flavors, coloring agents etc. The balance or the composition is water, which could advantageously be incorporated at levels of from 0.1-99.9%, more preferred 20-99%, most preferred 50 to 98% by weight.

[0064] The dispersions of the invention are useful as natural, non-caloric multifunctional ingredients in a wide range of food and pharmaceutical products. The aqueous phytosterol dispersions according to the invention can be used as hypocholesterolemic agents, as a non-calorie bulking agent, as a structuring and thickening material, as coloring, clouding and or opacity ingredients, as a high melting carrier for flavors, colors and other materials in a broad spectrum of food and pharmaceutical preparations. The phytosterols or other high melting lipids can also be used as high melting encapsulation materials.

[0065] Owing to their structuring functionality, the phytosterols and other high melting lipids can be used to replace fat structuring methods such as hydrogenation, interesterification, and use of natural hard fats such as tropical oils and/or animal fats. The aqueous phytosterol or high melting lipid dispersions can be used to replace conventional water structuring agents, as well, such as proteins, carbohydrates, gelatins and other thickeners and stabilizers. Eliminating partially hydrogenated fats removes trans fatty acids and reduces saturated fatty acids and calories. Moreover, the elimination of hydrogenated fats reduces the perception that the products are somehow "unnatural."

[0066] Shear can be generated in preparation of the dispersion of the invention by using, eg a turbo mixer, a colloid mill, a ball mill, a homogenizer or other mechanical or sonic devices.

[0067] The particle size measurements may be performed by using a Coulter LS particle size analyzer, ex. Coulter, Miami, FL or by Particle Sizing Systems Inc. Models 770 Accusizer and Nicomp 370, Santa Barbara, CA.

[0068] Preferably the phytosterols or other high melting lipids have a particle size of 15 microns or lower. Preferably, at least 90% and more preferably 100% of particle sizes fall within a range of between 10 nanometers and 50 microns.

[0069] Materials which are typically used include phytosterols either pure or technical grade, either in sterol or stanol form; saturated distilled mono and diglycerides, e.g. Myverol 18-04; water; and polysorbate 60 (Tween 60).

[0070] Unless stated otherwise or required by context, the terms "fat" and "oil" are used interchangeable herein. Where a phase is said to constitute essentially the entire product, it is meant that such phase constitutes at least 98 wt. %, especially more than 99 wt % of such product.

[0071] Where in this application phytosterols are mentioned, phytosterols, phytostanols, or mixtures thereof may be used as well. Likewise, where sterols are used in this application stanols are also contemplated. Thus, for instance, 4-desmethylsterols, 4-monomethylsterols and 4,4'-dimethylsterols, their stanol equivalents and mixtures thereof in any combination may all be useful.

[0072] Equipment which is typically employed includes Glass Beakers 250 ml and 2000 ml; hotplate and microwave oven; high shear turbomixer such as a Silverson with a fine screen gram scale, convention oven to melt phytosterol mix at about 150°C, and centrifuge.

Example I

Phytosterol Water Dispersion Process

[0073]

1. Melt 90g of phytosterols and 10g of Myverol 18-04 together in a 250 ml beaker.
2. Fill each of two 2000ml beakers with about 1500ml of water and heat to a boil in a microwave oven.
3. Place first 2000 ml beaker with the hot water on a hotplate set for about 95C which has been placed directly

below the Turbomixer agitator shaft.

4. Turn Turbomixer on and gradually increase shaft speed until water is turbulent and a good vortex has formed.

5. Slowly add molten (150C) 90/10 phytosterol/18-04 mixture to vortex and increase Turbomixer speed to submerge and quickly disperse the molten mix which will have a tendency to foam and set up quickly on the water's surface. Caution: Addition of molten 90/10 mix must be controlled to keep foaming to a minimum and the temperature mix must not drop by more than a few degrees during this step. Reheat 90/10 mix as necessary and maintain temperature of water at 95C by use of the hot plate.

6. After dispersing the first half of the 90/10 mix (about 50g) in the first beaker with 1500 ml of hot water continue to mix the phytosterol dispersion for about 2 to 3 minutes at high speed. Remove the first beaker from the Turbomixer and begin dispersing the remaining 90/10 mix in the second beaker containing 1500ml of hot water. Follow the same procedures and precautions.

7. Allow phytosterol dispersion to separate. Decant or siphon off water layer. Taste phytosterol layer; if bitterness exist combine the two phytosterol layers into one beaker and water wash five times using hot water and Turbomixer for each wash cycle. For each cycle, decant wash waster and use about 1500 ml of fresh hot water.

8. The washed or nonwashed phytosterol dispersion is concentrated in a centrifuge to a moisture level of about 70 to 75%. Store the concentrated phytosterol dispersion refrigerated.

9. A similar 90/10 phytosterol dispersion can be made by substituting 10g of polysorbate 60 for 10g of Myverol 18-04. Same procedure and precautions apply.

10. The dispersion process (particle size reduction) is facilitated by using 1% polysorbate 60 in the hot water to which the molten phytosterols are added under Turbomix agitation. However, use of polysorbate 60 in the water phase makes separation of the phytosterol layer more difficult and time consuming.

Typical Formulations Based on Water Dispersed Phytosterols:

[0074] In all examples the phytosterol applied (Phyto) was derived from soybean and consisted primarily of Beta-sitosterol (45%), campesterol (27%), stigmasterol (21%) and some minor other sterols (total 7%).

Example 2

65% Oil w/o spread with 10% phytosterols and no trans fats

Part I-Beaker with Oil Phase

[0075]

Sunflower Oil	260g
100% Phytosterols-pure technical	10g
	270g @ 150C

Part II-Beaker with Water Phase

[0076]

90/10 Phyto/18-04 Water Dispersion (72.5% Moisture)	122g @ 20C
Salt/Flavor/color	8g
	400g Cool to 30C
	and fill

[0077] Procedure: Add Part I to Part II under Turbomixer agitation. Cool w/o emulsion in a cold water bath (larger beaker) while emulsion is under agitation. Cool down to about 30C and fill in to cup. Refrigerate.

Example 3

65% Oil Duplex Emulsion Spread with 10% Phytosterols & no trans fats

Part I - Beaker with o/w Emulsion

[0078]

Sunflower Oil	70g @ 20/25C.
90/10 Phyto/18-04 Water Dispersion	121 g @ 20C.
Salt	8g
Polysorbate 60	1g
	200g @ 20/25C.

Part II - Beaker with External Oil Phase

[0079]

Sunflower Oil	198g @ 150C.
Myverol 18-04	2g @ 150C.
100% Phytosterols - pure/technical	10g @ 150C.
Flavor/Color qs add *	
	400g

* add flavor/color when Part I & Part II are being combined

[0080] Procedure: Using Turbomixer, prepare Part I by dispersing SF Oil in the 90/10 Phyto/18-04 dispersion in which the Polysorbate 60 has been well incorporated. Then add the Part I o/w emulsion to the beaker containing Part II again by using the Turbomixer. Use a cold water bath to cool this fat continuous emulsion down to about 30C. Fill into cups. Refrigerate.

Example 4

[0081] Formulations for 0%, 6& and 24% fat water continuous spreads with 10% phytosterols:

Ingredients	0%	6%	24%
90/10 PHYTO/18-04 (72.5% moisture)	162	162	162
SF oil	----	24	96
Water (95C)	215.95	193.15	122.45
Gelatin (beef)	5	4.5	4
Starch, Remyline AP	5	4.5	4
Lactose	4	3.9	3.5

(continued)

Ingredients	0%	6%	24%
Lactic Acid	.3	.3	.3
K Sorbate	.05	.05	.05
Salt	6.5	6.5	6.5
Buttermilk Powder	1	1	1
Beta Carotene CWS 1%	.15	.15	.15
Flavor and Vitamins	.05	.05	.05
	400g	400g	400g

[0082] Preparation: Disperse gelatin and starch in the 90C water using the turbomixer. Then add remaining ingredients under agitation. Add flavors/vitamins and the 90/10 Phyto/18-04 dispersion last. Cool down to 20-25C pour in cups. Refrigerate.

Example 5

0% fat reduced calorie mayonnaise

[0083]

	Control	New
Titanium Dioxide	1.0	---
Vinegar 120 grain	4.5	4.5
Mustard Flour	0.5	0.5
Food Starch Modified	10.0	5.0
K Sorbate	0.1	0.1
Na Benzoate	0.1	0.1
Salt	2.0	2.0
Sugar	8.0	8.0
Beta Carotene	0.1	0.1
Natural Spice Flavor	0.2	0.2
Natural Egg Flavor	0.2	0.2
Phosphoric Acid	0.2	0.2
Water	73.1	39.1
90/10 PHYTO/18-04 @ 72.5% Moisture	---	40.0
	100.0	100.0

[0084] Preparation: Cook all ingredients other than the 90/10 Phyto/18-04 dipersion in starch cooker. Cool the cooked starch and add the phytosterol dipersion under mild agitation. Mill final mixture through a colloid mill.

[0085] Formula replaces modified food starch by 50% and use of the artificial food color titanium dioxide (whitening and opacity agent by 100%)

Example 6

Creamy Italian Dressing

[0086]

	Control	New
Soybean Oil	45.0	36.0
Water	40.4	11.0
Sugar	4.5	4.5
Vinegar 120 Grain	3.0	3.0
HVF Algin	0.2	0.1
Buttermilk Powder	2.2	1.0
Lactic Acid	0.3	0.3
Lemon Juice Conc	0.4	0.4
Salt	2.5	2.5
Minced Onion	0.2	0.2
Minced Garlic	0.5	0.5
Xanthan Gum	0.2	0.1
Red Bell Peppers	0.2	0.2
MSG	0.1	0.1
Spices	0.1	0.1
Polysorbate 60	0.2	---
90/10 PHYTO/18-04 Moisture 72.5%	---	40.0
	100.0	100.0

[0087] Preparation: Combine ingredients under agitation and process through colloid mill.

[0088] Formula eliminates use of polysorbate 60 and reduces use of food gums by 50%, buttermilk powder use by 50% and soybean oil usage by 20%.

[0089] It will be apparent that for commercialization the previously mentioned process steps would be upscaled to the appropriate process and equipment sizes, types and standards practiced in the particular or relevant food industry.

[0090] It should be understood of course that the specific forms of the invention herein illustrated and described are intended to be representative only as certain changes may be made therein without departing from the clear teachings of the disclosure. Accordingly, reference should be made to the following appended claims in determining the full scope of the invention.

Claims

1. An aqueous phase dispersion or suspension comprising

   a) one or more high melting lipids having a mean size of 15 microns or lower, and
   b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2.

2. The dispersion according to claim 1 wherein the high melting lipids have a mean size of 10 microns or less.

3. The dispersion according to claim 1-2, wherein the high melting lipids have a melting point within the range of 75-

200°C.

4. The dispersion according to claims 1-3 wherein the high melting lipids are selected from the group convisting of: phytosterols, phytosterolesters, sterols, sterolesters, stanol, stanolesters, wax-esters, hardened vegetable oils, saturated tryglyceride fractions of vegetable oils, mono- and digiycerides.

5. The dispersion according to claims 1-4, comprising 0.1 - 99 wt.% preferably 5-75 wt.%, more preferably 10-50 wt.% of the high melting lipids.

6. The dispersion according to claims 1-5 incorporated into a foodstuff selected from the group consisting of water-continuous spreads, fat continuous spreads, bicontinuous spreads, dressings, beverages, dairy products, milk, cheese, yogurt, non-dairy coffee whiteners, beverages, confections and ice cream.

7. The dispersion according to claims 1-5 wherein the emulsifier is selected from the group consisting of monoglycerides, diglycerides, polysorbates, sodium stearyl lactylate and polyglycerol esters.

8. The dispersion according to claims 1-5 wherein the emulsifier is an oil-in-water emulsifier.

9. The dispersion according to claims 1-5 wherein the w/w ratio of emulsifier to high melting lipid is less than 1:2.25, in particular less than 1:3.

10. The dispersion according to claims 1-5 wherein the high melting lipid is a phytosterol which is selected from the group consisting of Beta sitosterol, campesterol, stigmasterol, brassicasterol and ergosterol.

11. An aqueous phase dispersion or suspension according to claims 1-5, also comprising a non-high melting lipid in a w/w-ratio to high melting lipid of less than 1:6, preferably less than 1:8.

12. A water continuous edible spread according to claim 6 comprising a discontinuous fat phase constituting from 0 to 85 wt. % of said spread, and a continuous aqueous phase constituting from 15 to 100 wt. % of said spread.

13. The spread according to claim 12 wherein the aqueous phase comprises a non-phytosterol emulsifier, in a w/w ratio of said emulsifier to high melting lipid of less than 1:2.

14. The spread according to claims 12-13 wherein

a) the high melting lipids are phytosterols and have a mean size of 15 microns or lower, and
b) wherein the aqueous phase comprises a non-phytosterol emulsifier, and a non-high melting, non-phytosterol lipid, the w/w ratio of the non-phytosterol lipid, to the high melting phytosterol being less than 1:6.

15. The spread according to claims 11-14 wherein the fat phase ranges from 0 to 40 wt. % of said spread, and wherein the aqueous phase constitutes from 60 to 100 wt. % of the spread.

16. The water continuous spread of claims 11-15 wherein the aqueous phase includes from 5 to 50 wt. % high melting lipids.

17. An edible, fat continuous spread according to claim 6 comprising a continuous fat phase constituting from 15 to 80 wt. % of said spread and a discontinuous aqueous phase constituting from 20 to 85 wt. % of said spread.

18. The fat continuous spread according to claim 17 wherein the fat phase includes a structuring lipid selected from the group consisting of a) high melting lipids and b) lipids which are not high melting.

19. The fat continuous spread according to claims 17-18 wherein the structuring lipid is selected from the group consisting of hydrogenated oil, or interesterified oil, or fractionated oils, or non-hydrogenated, non-interesterified non-fractionated hardstock or hydrogenated hardstock fat, or interesterified hardstock fat, or hydrogenated hardstock fat.

20. The fat continuous spread according to claims 17-19 wherein the aqueous phase includes from 5 to 50 wt. % phytosterols.

21. The fat continuous spread according to claims 17-20 wherein the fat phase includes a structuring agent selected from the group consisting of hydrogenated and/or esterified phytosterols, free phytosterols, waxes in particular waxes from aliphatic long chain fatty acids.

22. The fat continuous spread according to claims 17-21 wherein the aqueous phase includes dispersed therein a further fat phase.

23. The fat continuous spread according to claims 17-22 wherein the continuous fat phase comprises phytosterols.

24. The fat continuous spread according to claims 17-23 wherein the continuous fat phase comprises from 0.5 to 5 wt. % high melting lipid based on the weight of the product, and the aqueous phase comprises from 2 to 15 wt. % high melting lipid based on the weight of the product.

25. A method for preparing an aqueous dispersion or suspension of high melting lipid comprising mixing together a molten high melting lipid, molten surfactant, and water under shear.

26. The method according to claim 25 wherein said shear is provided by a turbo mixer, a colloid mill, a ball mill or a homogenizer.

27. The method according to claims 25-26 further comprising concentrating the suspension or dispersion.

28. A process for making a spread comprising dispersing a liquid oil into an aqueous dispersion of high melting lipid to form a first dispersion and then dispersing said first dispersion into a high melting lipid-containing oil phase.

29. The process according to claim 28 wherein the continuous oil phase comprises 1-25 wt. % high melting lipids, the aqueous phase comprises 1-25 wt. % of one or more high melting lipid and the continuous oil phase comprises 25-95 wt. % of the spread and the internal oil phase comprises from 0 to 70 wt. % of the spread, the aqueous phase comprising 0 to 75 wt. % of the spread.

30. The dispersion according to claim 4 wherein the high melting lipid is a chemically modified sterol.

31. The dispersion according to claim 30 wherein the chemically modified sterol is β sitostanol.

32. A fat based food product comprising an aqueous dispersion or suspension including:

a) one or more high melting lipids having a mean size of 15 microns or lower and
b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2,

wherein the fat used in the product is a fat comprising at least 30 wt. % preferably at least 45 wt. % of PUFA rich triglycerides, calculated on the total weight of the fat present in the product.

33. A fat based food product comprising an aqueous dispersion or suspension including:

a) one or more high melting lipids having a mean size of 15 microns or lower and
b) a non-sterol emulsifier, the w/w ratio of emulsifier to high melting lipid in said aqueous phase being less than 1:2,

wherein the fat in the food product comprises butterfat, and the total amount of phytosterol is in the range of 5-15 wt. %.

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EUROPEAN SEARCH REPORT

Application Number
EP 98 20 2536

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X Y	CA 2 091 152 A (KABI PHARMACIA AB) 6 September 1994 * page 16, line 26-29; claims 1-13 * * page 19, line 6-23; examples 1,2,13 *	1-9, 25-27 10-24, 28-33	A23D7/00 A23D7/015 A23L1/24 A23L1/307 A23L2/52
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TECHNICAL FIELDS SEARCHED (Int.Cl.6)
A23D
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	1 December 1998	De Jong, E

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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T : theory or principle underlying the invention
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D : document cited in the application
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& : member of the same patent family, corresponding document

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
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(54) **Method of producing a flavour composition suitable for flavouring tea**

(57) A flavour composition suitable for flavouring tea is made by mixing an oily, foreign flavour with an aqueous tea extract or an aqueous solution of a dried tea extract to form an oil-in-water emulsion, followed by spray drying the resulting emulsion.

The flavour composition may be mixed with dried tea extract and/or sugar, or agglomerated with tea dust and mixed with leaf tea.

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SPECIFICATION

Method of producing a flavour composition suitable for flavouring tea

The present invention relates to a method of producing a flavour composition suitable for flavoring tea.

Tea flavoured with a foreign flavour, that is to say, an added flavour not naturally occurring in the tea, for example, of citrus fruit, is valued by large groups of consumers for its particular organoleptical properties.

Various products are commercially available from which tea with a desired foreign flavour can be prepared in a simple manner. Some of these products are mixtures of dried tea extracts, foreign flavour, for example citric acid, and possibly sugar, which when dissolved in water, to which ice cubes are added, produce so-called "iced tea". Other known poducts consist of flavoured leaf tea, from which a brew can be prepared in the manner conventional for leaf tea. Various methods are known for the preparation of such leaf tea. In some of these, an oily flavour is applied direct to the leaf tea, for example, by sprinkling the tea with the desired quantity of flavour and subsequently mixing it together, or by keeping the tea in agitation in a tumbler while the flavour is sprayed into the tea mass in the desired quantity. Although it is possible in this way to produce a product having organoleptical properties appreciated by many, these methods have the disadvantage that, in connection with the volatility of some components of the oily flavour, the tea thus flavoured must be packed in an air-tight fashion, and once the package has been opened, is apt to lose its particular flavour quite rapidly. Such an oily flavour deteriorates when contacted with oxygen from the air and, as a result, loses its valued organoleptical properties. As a consequence, these unstable products are less suitable for being packed in teabags.

In order to eliminate this disadvantage, it has been proposed to flavour leaf tea by mixing the tea with a flavour composition in which the flavour is encapsulated in a water-soluble, non-volatile carrier, so that the flavour cannot volatilize or deteriorate, even if the composition is exposed to the open air for a long time. Such a stable flavour composition can be prepared, in accordance with Dutch patent application 74,11619, by spray drying an emulsion of an oily flavour in an aqueous solution of gum arabic. The particles of the powder thus produced consist of microcapsules, in which the flavour is encapsulated within a skin of gum arabic. This flavour composition can be agglomerated with tea dust to a particle size suitable for being mixed with leaf tea. A similar method is proposed in Dutch patent application 76,11520. Although the flavoured tea thus produced can be packed and stored without particular precautions, for example in tea-bags without the need to fear that the flavour deteriorates or is lost through volatilization, these known flavour compositions have the disadvantage of containing a rather large proportion of foreign carrier, for example more than 20% gum arabic, which cannot be beneficial to the organoleptical properties of the product. In addition, the presence of foreign carriers in flavoured tea is objected to in certain countries on the ground of legal provisions.

It has now been found that it is possible to replace the foreign carrier in a flavour composition of the type described above by a carrier belonging to tea, namely, by soluble tea constituents. In particular it has been found that, by spray drying an emulsion of an oily flavour in an aqueous tea extract, a powdered product is produced, in which the oily part of the flavour in the particles is encapsulated in a skin of dry, water-soluble components, and furthermore that the flavour composition thus produced is highly stable, and lends itself excellently to agglomeration by means of water as the agglomerating liquid.

The present invention accordingly provides a method of preparing a flavour composition suitable for flavouring tea, which comprises mixing an oily foreign flavour with an aqueous solution of a carrier to form an oil-in-water emuslion, and spray drying the resulting mixture, said method being characterised by using as a carrier solution an aqueous tea extract or an aqueous solution of a dried tea extract.

For the preparation of the flavour composition, a flavour is used that is suitable for flavouring tea. Examples are lemon, orange, rum, peppermint, bergamot, jasmine and rose flavour. The flavour consists in full or in part of oily components immiscible with aqueous tea extracts. The content of oily components should be sufficient to form an emulsion thereof in an aqueous tea extract. In addition to the oil phase, the flavour may contain an aqueous phase. This aqueous phase is or is not homogeneously mixed with the oil phase, and may contain solid, for example citric acid, in solution.

The selected oily flavour is intimately admixed with an aqueous tea extract to form an oil-in-water emulsion. Naturally, instead of an extract, an aqueous solution of a dried tea extract may be used. The extract may be prepared in known manner by means of hot water from fermented or non-fermented (green) tea. The solid content of the emulsion should be suifficient for stabilization of the flavour, that is to say, that practically all flavour-containing oil droplets can be encapsulated within a skin of solid, water-soluble components. These solid, water-soluble components are the solid tea components from the extract, and, if the flavour used contained an aqueous phase with solid dissolved therein, the solid components from the flavour. Hitherto, the best results have been obtained using emulsions having solid levels in the aqueous phase of at least 20% by weight. The requirement that the mixture of flavour and extract must be capable of being spray dried imposes an upper limit on the solid level of the extract, which depends on the spray drying plant used. Mostly this upper limit is in the order of 50% by weight.

Preferably the mixture of flavour and extract is of such a composition that the spray dried mixture contains 5-35% components of the selected foreign flavour as an oil phase, depending on the strength of the flavour. In this connection, for example, lemon, orange and rum flavour are regarded as weak

flavours, and bergamot, jasmine and rose flavour as strong ones, peppermint flavour having a medium strength. In order to set off weak flavours to their advantage, they are preferably mixed with an extract of green tea, which has a tea flavour less pronounced than that of an extract of fermented tea. Strong flavours can also be combined with an extract of fermented tea.

The spray drying of the mixture of flavour and extract may be effected in a known manner.

The flavour composition produced by spray drying can be used in various manners. Thus, for example, it may be used as such or in admixture with dried tea extract (instant tea powder) and/or sugar for the preparation of so-called "iced tea". In order to improve its moisture-receptiveness, homogeneity and velocity of dissolution, the composition or mixture may be agglomerated. If an extract of green tea has been used for the preparation of the flavour composition, the natural tea flavour of the composition is sometimes insufficient, and blending with dried tea extract is desirable. This is mostly unnecessary if an extract of fermented tea has been used.

If the flavour composition is intended for flavouring fermented or non-fermented leaf tea, it is preferably agglomerated with tea dust. The tea dust may consist of one or more tea fractions collected in sieving dried fermented tea, for example, from so-called "siftings" having a particle size of 0.1-0.5 mm, or of a blend thereof with so-called "dust tea" having a particle size of 0.2-0.7 mm.

Agglomeration may be effected in known manner, for example, in an agglomeration dish as described in Dutch patent application 74,11619, referred to hereinbefore. Water is a suitable agglomeration liquid.

Preferably, the flavour composition is agglomerated with approximately the same quantity of dust tea by weight. By a suitable control of the conditions during the agglomeration, the particle size of the agglomerated product can be attuned to the dimensions of the leaf tea with which the granular flavour composition must be admixed.

Where necessary, the agglomerated product is dried and subsequently mixed with leaf tea. The quantity of flavour composition in the mixture depends on the strength of the flavour, and is generally 5-24% by weight.

The present invention will now be further illustrated by way of the following examples.

Example I

3.8 kg of a commercially available liquid lemon flavour having a water content of approximately 60% by weight, and comprising a mixture of deterpinated lemon oil and lemon juice inspissated to approximately 15% of its original volume was emulsified in a solution of 14.2 kg dried extract of green tea in 30 kg water, whereafter the resulting emulsion was spray dried. This resulted in 15.7 kg flavour composition, which by means of water was agglomerated with a like quantity of tea siftings to form granules having a size of 0.5-2mm. For the agglomeration, an agglomeration dish was used as described in Dutch patent application 74,11619.

8 kg of the agglomerated product was mixed with 92 kg fermented leaf tea having sizes of 0.5-4mm, whereafter the mixture was packed in teabags of wetstrength paper. The tea thus flavoured produced a tea drink with the desired lemon flavour even after prolonged storage without particular precautions.

Example II

6 kg of the liquid lemon flavour described in Example I was emulsified in 40 kg aqueous extract of fermented tea having a dry content of 14% by weight, whereafter the resulting emulsion was spray dried to produce 8 kg flavour composition. By intimately mixing 19 g of this flavour composition with 90 g powdered sugar, an instant powder was produced, which when dissolved in ice water to a concentration of 10 g/l produced so-called "iced-tea".

Example III

10 kg of a commercially available peppermint oil was emulsified in 40 kg aqueous extract of fermented tea having a dry content of 14% by weight, whereafter the resulting emulsion was spray dried. The product was 8 kg flavour composition, which by means of water was agglomerated with a like quantity of tea siftings to form granules having a size of 0.2-2.5 mm. A mixture of these granules with leaf tea as in Example I was perfectly stable and suitable for being packed in teabags. The tea thus flavoured produced a tea drink having the desired peppermint flavour.

CLAIMS

1. A method of preparing a flavour composition suitable for flavouring tea, which comprises mixing an oily foreign flavour with an aqueous solution of a carrier to form an oil-in-water emulsion, said carrier solution being an aqueous tea extract or an aqueous solution of a dried tea extract, and spray drying the resulting mixture.

2. A method as claimed in claim 1, in which the solid content of the aqueous carrier solution is selected so that the emulsion contains at least 20% by weight of dry components dissolved in the aqueous phase.

3. A method as claimed in claim 1 or claim 2, in which the aqueous carrier solution comprises up to 50% by weight of solids.

4. A method as claimed in any of claims 1 to 3, in which the flavour and the aqueous carrier solution are mixed together in such a ratio that the spray dried product contains 5-35% by weight of foreign flavour components as an oil phase.

5. A method as claimed in any of claims 1 to 4, in which the oily foreign flavour is lemon, orange, rum, peppermint, bergamot, jasmine or rose flavour.

6. A method as claimed in any of claims 1 to 5, in which the spray dried product is agglomerated with tea dust.

7. A method as claimed in claim 6, in which water is used as an agglomeration liquid.

8. A mixture of dried leaf tea and a flavour composition produced by the method of claim 6 or

claim 7.

9. A teabag of wet-strength paper, filled with a mixture according to claim 8.

10. A method as claimed in claim 1, and substantially as hereinbefore described with referene to any of the Examples.

11. A mixture as claimed in claim 9 and substantially as hereinbefore described with reference to Example I or Example III.

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